Agilent 1260 Infinity Analytical SFC System

User Manual



Agilent Technologies

Notices

© Agilent Technologies, Inc. 2010 - 2012

No part of this manual may be reproduced in any form or by any means (including electronic storage and retrieval or translation into a foreign language) without prior agreement and written consent from Agilent Technologies, Inc. as governed by United States and international copyright laws.

Manual Part Number

G4301-90001

Edition

12/2012

Printed in Germany

Agilent Technologies
Hewlett-Packard-Strasse 8
76337 Waldbronn

This product may be used as a component of an in vitro diagnostic system if the system is registered with the appropriate authorities and complies with the relevant regulations. Otherwise, it is intended only for general laboratory use.

Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

Technology Licenses

The hardware and/or software described in this document are furnished under a license and may be used or copied only in accordance with the terms of such license.

Restricted Rights Legend

If software is for use in the performance of a U.S. Government prime contract or subcontract, Software is delivered and licensed as "Commercial computer software" as defined in DFAR 252.227-7014 (June 1995), or as a "commercial item" as defined in FAR 2.101(a) or as "Restricted computer software" as defined in FAR 52.227-19 (June 1987) or any equivalent agency regulation or contract clause. Use, duplication or disclosure of Software is subject to Agilent Technologies' standard commercial license terms, and non-DOD Departments and Agencies of the U.S. Government will receive no greater than Restricted Rights as defined in FAR 52.227-19(c)(1-2) (June 1987). U.S. Government users will receive no greater than Limited Rights as defined in FAR 52.227-14 (June 1987) or DFAR 252.227-7015 (b)(2) (November 1995), as applicable in any technical data.

Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

In This Book

This Manual discribes all about the Agilent 1260 SFC System.

1 Introduction to Supercritical Fluid Chromatography (SFC)

This chapter provides an overview of the history, theory and benefits of SFC.

2 Site Requirements and Specifications

This chapter provides information on environmental requirements, physical and performance specifications only for the G4309 Agilent 1260 Infinity Analytical SFC System.

3 Installing the G4309A Agilent 1260 Infinity SFC System

This chapter provides an overview of the installation and setup of the hardware and software

4 Configuring the System

How to configure the Agilent 1260 Infinity SFC Analytical system and Agilent 1260 Infinity SFC Control Module in ChemStation.

5 Using the Agilent 1260 Infinity SFC Control Module

This chapter provides information and hints on the use of the SFC System.

6 Maintenance and Repair

In this chapter only the SFC specific procedures are described. For procedures similar to the Agilent module procedures, please refer to the single module manuals (G1312C, G1329B, G1316C, G1315/65C, G4225A)

7 Parts for Maintenance

This chapter provides information on parts for maintenance and repair.

8 Identifying Cables

This chapter provides information on cables used with the Agilent 1200 Infinity Series modules.

9 Appendix

This chapter provides addition information on safety, legal and web.

Contents

1 Introduction to Supercritical Fluid Chromatography (SFC) 7

History of SFC 8
Theory of SFC 9
Benefits of SFC 10
Common flow path overview for packed column SFC instrumentation 11
The Agilent 1260 Infinity Analytical SFC System 12

2 Site Requirements and Specifications 23

Site Requirements 24
Specifications 28

3 Installing the G4309A Agilent 1260 Infinity SFC System 39

Hardware Installation 40
Software Installation 75

4 Configuring the System 79

Configuring SFC interface in ChemStation 80
Setting up the Method 84
Status 86
Control 87
Testing the System 88

5 Using the Agilent 1260 Infinity SFC Control Module 89

Powering up the Module 90
Power-up Sequence and Operational Control States 91
Operational Control States 92
Controlling the Agilent 1260 Infinity SFC Control Module through the Agilent ChemStation 95
Running a method on the SFC system 98
Shutting Down the SFC System 101

6 Maintenance and Repair 105

Inspection and Preventative Maintenance Intervals 106
General Maintenance procedures 108

7 Parts for Maintenance 119

Agilent 1260 Infinity SFC Control Module Parts 120
Agilent Module Parts 121

8 Identifying Cables 125

SFC Control Module Cables 126
Overview 127
BCD Cables 128
External Contact Cable 130
CAN/LAN Cables 131
RS-232 Cables 132

9 Appendix 133

General Safety Information 134
The Waste Electrical and Electronic Equipment (WEEE) Directive (2002-96-EC) 137
Radio Interference 138
Sound Emission 139
Solvent Information 140
Agilent Technologies on Internet 141

1 Introduction to Supercritical Fluid Chromatography (SFC)

History of SFC 8
Theory of SFC 9
Benefits of SFC 10
Common flow path overview for packed column SFC instrumentation 11
The Agilent 1260 Infinity Analytical SFC System 12
- The Agilent 1260 Infinity SFC Control Module (G4301A) 13
- HPLC-SFC binary pump (G4302A) 13
- SFC-Autosampler (G4303A) 14
- The Column Compartment (G1316C) 21
- UV-detection (DAD G1315C and MWD G1365C) 21
- Applications 21
- Columns 22

This chapter provides an overview of the history, theory and benefits of SFC.

History of SFC

Supercritical fluid chromatography (SFC) was first introduced by Klesper et al. in 1962 (Klesper, E.; Corwin, A. H.; Turner, D. A. J. Org. Chem. 1962, 27,700.) for the separation and analysis of a porphyrin mixture using open tubular SFC. The first commercial instruments using packed columns were available from Hewlett-Packard (HP) in 1982. Since then, several vendors have developed and commercialized packed column SFC instrumentation for analytical as well as for preparative separation. SFC is widely accepted for the separation of chiral compounds and increased user interest has been observed for a wide spectrum of small to medium sized molecules due to the analysis speed achieved and the low solvent consumption.

The latest introduction of analytical SFC instrumentation, the Agilent 1260 Infinity SFC Control Module coupled to an Agilent 1260 Infinity Binary LC system optimized for SFC.

Theory of SFC

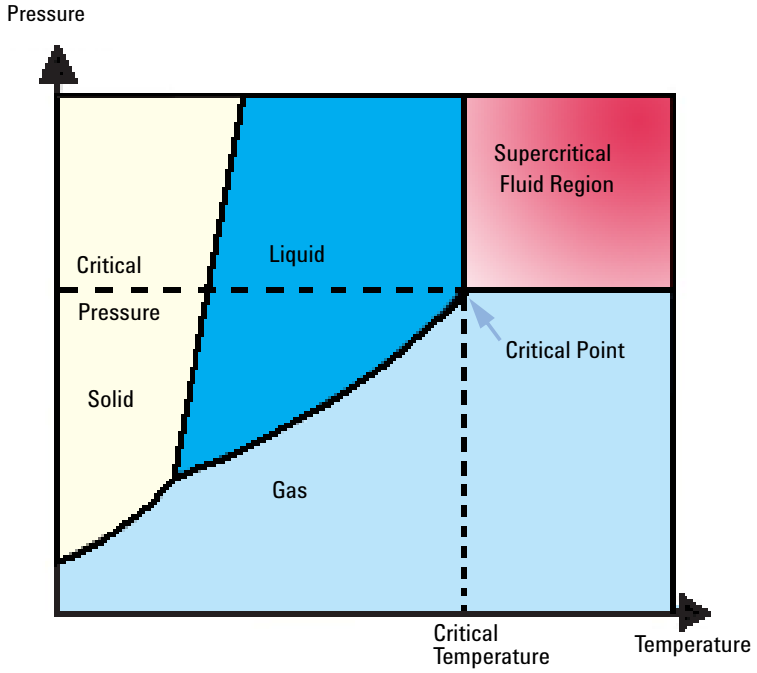


Figure 1 State of a solvent

The superior separation properties achieved by SFC can be explained best by the thermodynamics of liquids and gases (see the phase diagram in Figure 1 on page 9). Above a critical pressure, liquids can no longer enter the gaseous state; similarly, above a critical temperature, gases cannot be converted to liquids. Above both the critical pressure and temperature (characterized by the critical point), solvents are in the supercritical state. Under these conditions, the mobile phases exhibit gaseous as well as liquid-like properties. *The major advantages of this state related to chromatography are improved diffusion characteristics and mass transfer and low viscosity, which result in high separation efficiency and fast separation capability.*

Benefits of SFC

SFC is widely accepted for the analysis and separation of chiral compounds. In addition, it gains increasing acceptance as a complementary liquid-based separation technique to HPLC for high-throughput and high-resolution analysis of complex mixtures. This is due to the thermodynamic properties of supercritical fluids, which can be exploited for high throughput and high efficiency. In addition, the mild thermal conditions also allow for the analysis of thermally labile compounds. Typically, analysis times and column re-equilibration are decreased by a factor of 3–5 compared to standard HPLC. With the increasing costs of organic solvents and the environmental awareness to minimize toxic waste, production SFC is increasingly accepted as the “green alternative” to normal phase or reversed phase chromatography, gaining popularity in method development and UV- and MS-based separation and purification. A variety of parameters, such as stationary phase selection, mobile phase composition, modifier type and concentration, column temperature and system pressure, can be easily manipulated to fulfill separation requirements by influencing, optimizing and exploiting selectivity in SFC.

Common flow path overview for packed column SFC instrumentation

In commercially available SFC systems, CO_2 is initially pumped in liquid state and is brought into the supercritical state by heating it above the critical temperature before it enters the high-pressure area of the LC instrument. After high-pressure mixing with a modifier, the mobile phase passes through the injection loop, where the sample is introduced into the supercritical stream, and further transported to the separation column. The high pressure of the mobile phase must be maintained downstream of the detector outlet using a backpressure regulator to keep the mobile phase in its supercritical condition over the complete flowpath.

The Agilent 1260 Infinity Analytical SFC System

The Agilent 1260 Infinity Analytical SFC System (G4309A) consists of a binary HPLC-SFC pump (G4302A), a degasser (G4225A), an SFC Autosampler (G4303A), a thermostatted column compartment (G1316C), and either a DAD (G1315C) or MWD (G1365C) equipped with a high pressure SFC flow cell and the SFC control module (G4301A). The SFC control module, redestills and preconditions the CO_2 by boosting the pressure to just below the column head pressure, relieving the HPLC-SFC pump of any compression requirements. This results in low detector noise and significantly higher sensitivity. Therefore, the Agilent HPLC-SFC pump receives pre-conditioned CO_2, and acts only as metering device for the mobile phase flow and to form the gradient with the second pump head by adding the appropriate amount of modifier solvent. Downstream of the detector, the mobile phase is redirected back into the SFC control module to an integrated back pressure regulator that maintains the backpressure over the system. The Agilent 1260 Infinity SFC system is completely controlled by Agilent ChemStation software. The system diagram is shown below:

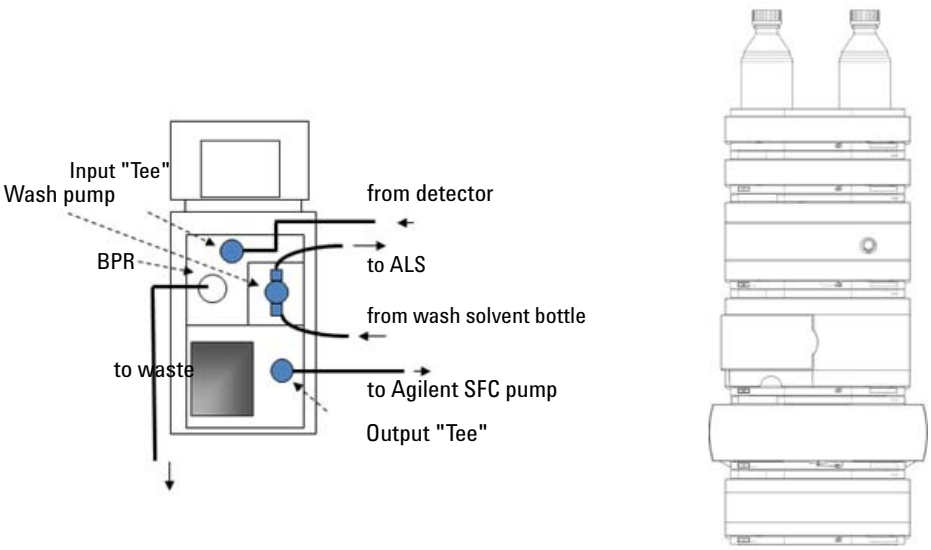


Figure 2 Agilent 1260 Infinity Analytical SFC System

The Agilent 1260 Infinity SFC Control Module (G4301A)

The Agilent 1260 Infinity SFC Control Module is responsible for all tasks connected to pre- and post-conditioning of the mobile phase. In contrast, flow rate, mobile phase composition, detection, column temperature and data analysis are controlled by the modules of the Agilent 1260 Infinity SFC system in combination with the ChemStation software. This includes metering the carbon dioxide flow and mixing the modifier into the mobile phase by the HPLC-SFC binary pump.

In detail, the Agilent 1260 Infinity SFC Control Module uses vapor-phase carbon dioxide, redestills it to the liquid state and boosts its pressure to just under the column head pressure. Since the CO_2 gas is a very poor solvent, most contaminants in the carbon dioxide are left in the source, which allows for the use of inexpensive, beverage-grade CO_2, unlike in any other commercially available instrument. The SFC control module further recollects the effluent from the UV (or other) detector and controls the backpressure up to 400 bar over the complete system. In addition, the SFC control module delivers the wash solvent to flush the fixed (sample) loop of the SFC autosampler.

HPLC-SFC binary pump (G4302A)

The HPLC-SFC binary pump is equipped with passive inlet valves and with special seals and pistons to allow for CO_2 pumping in channel A while channel B adds organic modifier for either isocratic or gradient performance. Pumphead B is also equipped with a purge valve to allow for quick changeover of the organic modifier.

SFC-Autosampler (G4303A)

In SFC, the complete solvent flow path needs to be pressurized under all conditions to avoid expansion of the supercritical fluid. This excludes the use of a variable injection loop design that is generally used in other Agilent autosamplers. Therefore, the autosampler used in the Agilent 1260 Infinity analytical SFC system has been converted to a fixed loop injector containing a 5 µL sample loop.

The injection loop of the autosampler is installed between two ports of the 2 position/6 port injection valve. The total delay volume of the injector is about 3.3 µL. As with any fixed loop injector, overfill of sample is necessary to inject 5 µL reproducibly. To completely fill the sample loop, an excess of sample is required. This is about 3 loop volumes (15 µL of the installed loop) of sample to achieve 95 % of the maximum loop volume. For smaller injection volumes, the loop needs to be partially filled by sandwiching the sample between two air bubbles, one on either side, followed by a plug of modifier or other solvent behind the sample. Default methods for full loop filling are provided, and should be used as initial injection conditions. The default methods are automatically loaded in ChemStation.

The recommended temperature setting on the left heat exchanger in order to achieve minimum noise is typically between 37 °C and 40 °C for G1315C or G1365C detectors using the 10 mm flow cell. This needs to be optimized empirically.

Standard ALS configuration: two groove rotor

Most Agilent automated liquid samplers (ALS) use the broken loop mode of injection with a 2-groove rotor. One groove alternately connects the pump to either the column or to the back of the metering syringe. The second groove alternately connects the needle port to either the waste port or to the column.

Loading position (or Bypass position in CS)

In the first position, (pump to column; needle port to waste) the metering device in the automated liquid sampler is filled with the mobile phase at atmospheric pressure. The back of the device (inlet) is *dead-headed* against the rotor in the injection valve.

The metering device can then withdraw sample from a vial or air from the sample compartment to create segmented flow. The sample is stored in the

needle and, for large injections, in the tubing in the arm upstream of the needle. The rotor, piston seal and all must be in good shape, leak tight and properly maintained for accurate metering and aspiration of the sample.

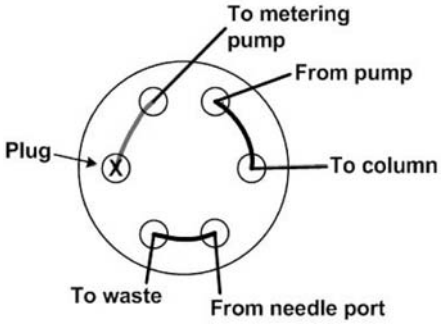


Figure 3 Loading position

Injection position (or MainPass position in CS)

The maximum injection volume with the standard metering device is 100 µL. The piston is driven by a stepper motor. Full travel on the piston is accomplished using thou-sands of motor steps. Each step results in a displacement of ~0.01 µL, and reproducibility could approach, or be better than, 0.1 %.

If all injector components are leak tight, the accuracy of the metering device is the only component that should affect the reproducibility of injections. A leaky needle port, a worn rotor or stator in the valve, or a worn piston seal in the metering device are the most likely causes of poor precision, other than leaking fittings.

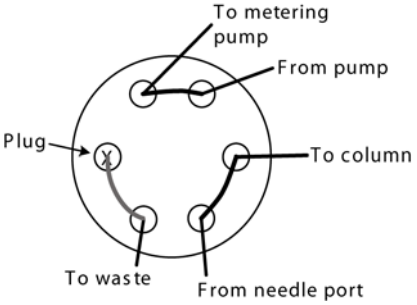


Figure 4 Injection position

Carryover

Carryover is largely a function of the cleanliness of the outside of the needle, or of a gap between the rotor and stator caused by wear. In some models, needle wash is provided to wash the outside of the needle between injections.

When the injection valve is switched to position two (pump to metering device; needle port to column, Inject or MainPass), the mobile phase passes through the metering device, down the tubing, through the needle and port and into the column. The mobile phase pushes the sample out of the needle/tube and washes these devices out.

There is no wasted sample. All of the sample withdrawn from the sample vial is injected. The valve is in this position most of the time and is only switched to position one (Load or Bypass), to load sample. The inside of the tube and needle, needle port, and valve body are all nearly continuously washed with fresh mobile phase during the run.

ALS modification for SFC: using the three-groove rotor

In SFC, the two-groove rotor is replaced with a three-groove rotor, and an external loop is added to the injection valve.

- The needle port is directed to enter either the inlet of the external loop or to waste.
- The waste port is directed to either the outlet of the loop or to the needle port.
- The pump is directed to either the inlet of the sample loop or to the column.
- The column is directed to either the outlet of the sample loop or to the pump.

The inlet of the metering device is no longer connected to the injection valve but is, instead, connected to an external wash pump. The metering pump should always be completely filled with the wash solvent, with no air bubbles.

Loading Position

The first position (pump to column, needle port to loop inlet, waste port to loop outlet) corresponds to the HPLC two-groove rotor load position. The needle is used to withdraw sample from the sample vial and air from the compartment in exactly the same way as for HPLC. The same metering device

is used and driven at the same speed with the same accuracy and precision. The needle is then positioned in the same needle port just as it is for HPLC.

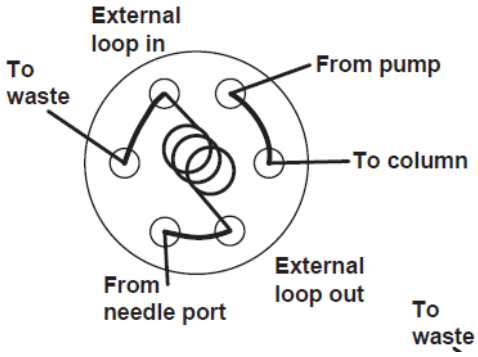


Figure 5 Loading position

Full loop injections

For full loop injections, the amount of sample withdrawn is actually substantially larger than the volume of the loop. This volume is normally two to three times greater than the sample loop's volume.

Most of this larger volume is pushed through the loop, overfilling it. The physical volume of the loop is primarily what determines the precision of the injection and the actual amount injected. The accuracy of the metering device is of secondary importance.

Overfilling the loop guarantees that the sample is a *midstream* sample without the dilution found on the ends caused by the parabolic flow profile of the sample as it moves through the tubing. Compared to the standard approach, the external loop mode wastes sample when maximum precision is required. However, reproducibility can be identical in both cases.

Modifying the Agilent ALS for SFC adds an additional step when setting up injections. The needle port and its connecting tubing have approximately 2 µL of dead volume after the needle and before the sample loop. In HPLC (two-groove rotor) this tube is flushed with mobile phase during injection.

In SFC this tube is no longer exposed to flowing mobile phase and any sample left in the tubing during injection will be lost to waste. Additionally, if this dead volume loss is not accounted for, 2 µL of sample will not be injected. For

example, the metering device delivering 5 µL of sample may only result in 3 µL of sample making it into the sample loop.

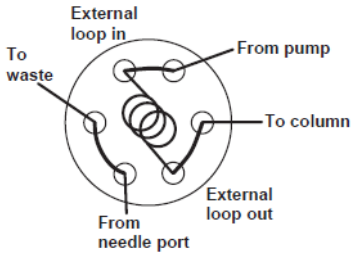


Figure 6 Injection position

Partial loop injections

For partial loop injections with the three-groove rotor, the metering device accuracy is more important. Accuracy and reproduci-bility are determined by the metering device, not by the loop volume. The most effective way to accomplish a partial loop injection is to:

- include two air gaps – one before and one after the sample plug. Full fixed loop injection also has these two air gaps to prevent sample loss as the needle moves out of the sample vial and into the needle port
- use a careful choice of air volumes to position the sample plug in the middle of the loop

The air gaps are extremely effective in controlling the shape and volume of the sample plug. Small air bubbles do not affect the chromatography.

A detailed procedure on how to use the Agilent 1260 Infinity SFC system for reproducible partial loop injection is described in Technical Note "*Injecting variable volumes using the partial loop fill method with the Agilent 1260 Infinity analytical SFC System*" Agilent Technical Overview 5591-0514EN on www.agilent.com/chem/sfc.

Carryover

For partial loop injections with SFC, the sample is exposed to the inside and the outside of the needle, the needle port, two grooves, and the sample loop. One groove and the sample loop are washed by the mobile phase. The other

two grooves are washed by the wash pump. The fittings in the injection system must be leak-tight to prevent drawing air bubbles into the metering device.

Both the two-groove (HPLC) and the three-groove (SFC) rotor settings have six potential leak spots in common. However, the three-groove setting has one additional leak spot. If the sample sits in the loop for any extended period of time, there is the potential for a leaky fitting causing siphoning. The waste line may be filled with solvent and sample.

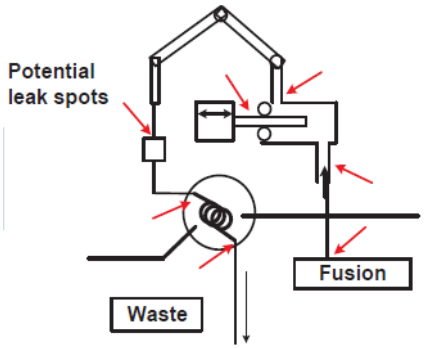


Figure 7 Potential leak spots in the loop, waste line, and the low pressure side of the injection system

If the waste bottle is positioned substantially below the injection valve, gravity can generate a 1 – 2 psi (0.1 bar) pressure differential between the metering device and the waste bottle causing the system to siphon if there is a leaky fitting on the low pressure side of the injection system (metering device, arm, needle, needle port, loop). If the wash solvent is stored in the solvent cabinet on top of the Agilent stack, gravity can generate an additional 1 – 2 psi of pres-sure on the wash pump.

The outlet check valve on the wash pump has a 15 psi spring pushing the ball into the seat. If this check valve is damaged, there is potential for wash solvent siphoning through the wash pump then through the metering device, arm, needle, and needle port, displacing some of the sample from the loop and causing loss of precision.

A quick check for this condition is simply to put the wash solvent bottle on the bench below the wash pump. This removes the pressure differential and the siphoning will stop.

Troubleshooting

Conversion of the ALS two-groove rotor to a three-grove rotor for SFC can only negatively affect reproducibility and carryover under the following conditions:

1 The user is NOT running the supplied injector program.

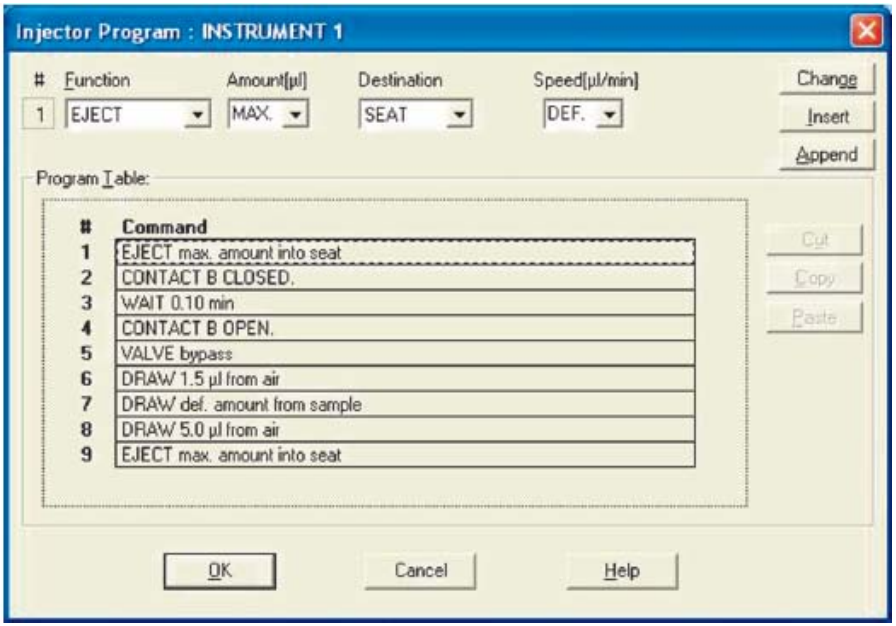


Figure 8 Injector program

2 The user is running a standard *broken loop* injection method.

3 A two-groove rotor is being used.

4 The tube that connects the wash pump in the SFC control module to the inlet of the metering pump (syringe) in the Agilent ALS is not leak tight, allowing the metering device to aspirate air.

5 The other end of the tube is not tight, allowing the syringe to aspirate air.

6 The wash pump is not delivering. To check: turn on the wash pump for 30 s and see if liquid drips out of the waste line from the injection valve.

7 The check valve on the outlet of the wash pump is leaking, allowing solvent to siphon.

The Column Compartment (G1316C)

The temperature of the mobile phase prior to detection is a critical parameter for minimizing baseline noise recorded in the detector flow cell. The heat exchanger on the right side of the column compartment is used to pre-heat the mobile phase before it enters the column, indirectly heating the column. The heat exchanger on the left side is used to change the temperature of the mobile phase to achieve minimum noise. This is of crucial importance, since the refractive index of carbon dioxide responds up to 50 times stronger than water-based mobile phases; thus, even small changes in temperature can significantly affect noise levels. Temperature changes of mobile phase with the left side heat exchanger e.g. between 38 °C and 49 °C resulted in a variation in peak-to-peak noise of over an order of magnitude.

The recommended temperature setting on the left heat exchanger in order to achieve minimum noise is typically between 37 °C and 40 °C for G1315C or G1365C detectors.

UV-detection (DAD G1315C and MWD G1365C)

The system can be equipped either with a DAD (G1315C) or an MWD (G1365C) using a high pressure detector flow cell suitable for SFC (10 mm path length, 13 µL volume), with short transfer tubing to minimize peak broadening. Electronic temperature control provides highest baseline stability and stable sensitivity values under fluctuating temperature and humidity conditions. This feature aids greatly in minimizing detector noise, and now enables impurity and EE (enantiomeric excess) analysis by SFC, particularly when using elevated temperatures.

Applications

SFC has gained a wide interest and acceptance in many small molecule applications because of its high separation speed and efficiency, selectivity, low operating costs, and due to low generation of organic solvent waste.

Important applications have been developed for the analysis of pharmaceutical drugs, natural products, fatty acids, vitamins, pesticides, lipids and chiral compounds. See Figure 9 on page 22.

Columns

In contrast to reversed phase separation, there is no universal stationary phase available for SFC separations. Most typically used stationary phases are ethyl pyridine, diol, cyano, amino, Silica and SCX columns. This usually leads to additional effort to screen different columns in order to achieve optimum separation. On the other hand, it provides a valuable tool for achieving different selectivities for a given analyte mixture. A review of column developments for SFC was recently published by *T.Berger, B.Berger & R.E.Majors* in LCGC North America, May 1, 2010

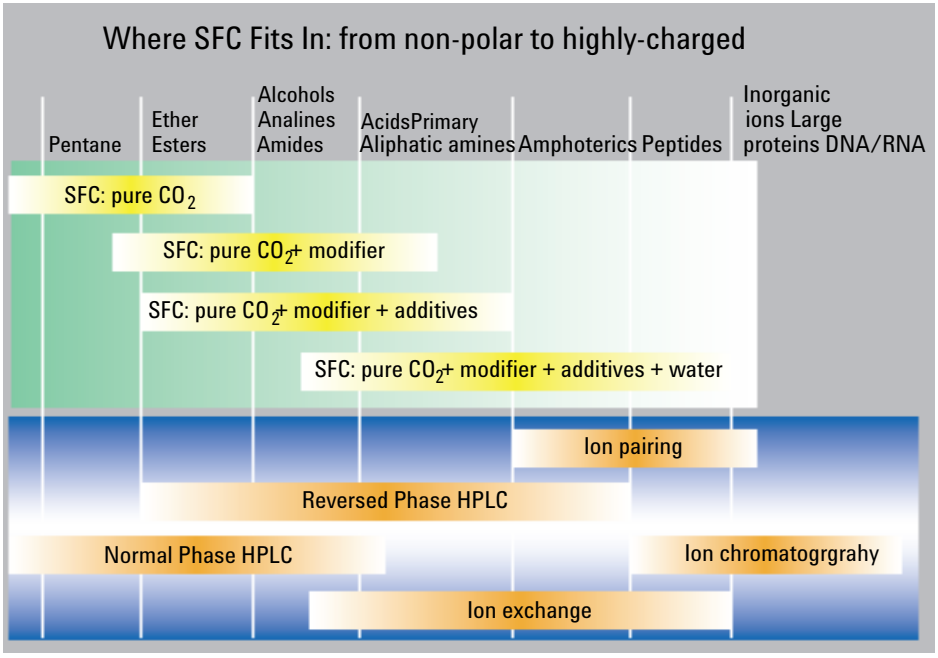


Figure 9 Where SFC Fits In: from non-polar to highly-charged



2
Site Requirements and Specifications

Site Requirements 24
- Power Considerations 24
- Power Cords 25
- Bench space 26
- Environment 27
- Ventilation 27

Specifications 28
- System Specifications 28
- Specifications 28
- Agilent 1260 Infinity System 32

This chapter provides information on environmental requirements, physical and performance specifications only for the G4309 Agilent 1260 Infinity Analytical SFC System.

Site Requirements

A suitable environment is important to ensure optimal performance of the instrument.

Power Considerations

The module power supply has wide ranging capability. It accepts any line voltage in the range described in Table 5 on page 33. Consequently there is no voltage selector in the rear of the module. There are also no externally accessible fuses, because automatic electronic fuses are implemented in the power supply.

WARNING

Hazard of electrical shock or damage of your instrumentation

can result, if the devices are connected to a line voltage higher than specified.

➔ Connect your instrument to the specified line voltage only.

WARNING

The module is partially energized when switched off, as long as the power cord is plugged in.

Repair work at the module can lead to personal injuries, e.g. electrical shock, when the cover is opened and the module is connected to power.

➔ Always unplug the power cable before opening the cover.

➔ Do not connect the power cable to the instrument while the covers are removed.

CAUTION

Inaccessible power plug.

In case of emergency it must be possible to disconnect the instrument from the power line at any time.

➔ Make sure the power connector of the instrument can be easily reached and unplugged.

➔ Provide sufficient space behind the power socket of the instrument to unplug the cable.

Power Cords

Different power cords are offered as options with the module. The female end of all power cords is identical. It plugs into the power-input socket at the rear of the module. The male end of each power cord is different and designed to match the wall socket of a particular country or region.

WARNING

Absence of ground connection or use of unspecified power cord

The absence of ground connection or the use of unspecified power cord can lead to electric shock or short circuit.

➔ Never operate your instrumentation from a power outlet that has no ground connection.

➔ Never use a power cord other than the Agilent Technologies power cord designed for your region.

WARNING

Use of unsupplied cables

Using cables not supplied by Agilent Technologies can lead to damage of the electronic components or personal injury.

➔ Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

Bench space

Agilent 1260 Infinity SFC Control Module

The Agilent 1260 Infinity SFC Control Module requires approximately 1 foot of linear bench space immediately adjacent to the target Agilent 1260 Infinity system stack. Approximately 5 inches of free space is required behind the instrument for cable access and adequate air flow for ventilation. Similar access to the rear of the 1260 Infinity system is also required to install cables and interface cards. As mentioned earlier, for optimal performance, the rear air space should not be heated significantly above room temperature by the exhaust of other instrumentation in the lab; rather, hot exhaust should be vented or directed upward from the instrument.

The module is designed to be installed on either side of the 1260 Infinity system stack with sufficient high pressure transfer tubing to attach to a double-stacked system. If the 1260 Infinity system is attached to a split-flow detector such as Mass Spec or ELSD, the Agilent 1260 Infinity SFC Control Module should be positioned on the opposite side of the stack. Shelves overhanging the Agilent 1260 Infinity SFC Control Module should provide a minimum of 6 inches of clearance to allow access to the rear power switch. Finally, the PC system interface to the Agilent 1260 Infinity SFC Control Module is USB 2.0. A six-foot cable is supplied with the system. The CPU must be placed within range of this cable. Alternately, the user may supply an extended length USB cable not to exceed 16 feet.

While the Agilent 1260 Infinity SFC Control Module can exist on either side of the 1260 Infinity system, it is often easier to locate it on the left side. For more specification details see “Agilent 1260 Infinity SFC Control Module” on page 28

Agilent modules

The dimensions and weight of your module (see “Agilent 1260 Infinity System” on page 32) allow it to be placed on almost any laboratory bench. It needs an additional 2.5 cm (1.0 inches) of space on either side and approximately 8 cm (3.1 inches) at the rear for the circulation of air and electric connections.

The module should be operated in a horizontal position.

If a Thermostatted Autosampler is installed, an additional 25 cm (10 inches)of space on either side for the circulation of air, and approximately 8 cm (3.1 inches) at the rear is required for electrical connections.

If a complete 1260 Infinity system is to be installed on the bench, make sure that the bench is designed to carry the weight of all the modules. For a system including the Thermostatted Autosampler it is recommended to position the modules in two stacks.

Environment

CAUTION

Condensation within the module

Condensation will damage the system electronics.

➔ Do not store, ship or use your module under conditions where temperature fluctuations could cause condensation within the module.

➔ If your module was shipped in cold weather, leave it in its box and allow it to warm slowly to room temperature to avoid condensation.

Ventilation

WARNING

Waste tube has to be connected to hood or vent

➔ The effluent from a supercritical fluid chromatograph may contain vaporized, toxic solvents. Never vent into an enclosed, occupied space. Always vent into a fume hood or vent to the outside.

Specifications

System Specifications

Type	Specifications
Flow range	0.1 mL/min to 5 mL/min (settable), 1 mL/min to 5 mL/min (recommended)
Maximum operating pressure	600 bar
Upgrade possibility of exisiting 1100/1200/1260 LC	Yes
Option for SFC/UHPLC in one system	Yes
Unattended operation	Leak sensors, diagnostic software features

Agilent 1260 Infinity SFC Control Module

Select the laboratory bench space before your system arrives. Pay special attention to the total height requirements. Avoid bench space with overhanging shelves. Pay special attention to the total weight of the modules and solvents you have in addition to the Agilent 1260 Infinity SFC Control Module. Make sure that your laboratory bench can support this weight.

WARNING

Personal injury

The Agilent 1260 Infinity SFC Control Module module is heavy.

➔ Enlist the aid of a co-worker to share the lifting load to avoid personal injury.

Table 1 Physical Specifications (G4301A)

Type	Specification
Weight	26 kg 56 lbs
Dimensions (height × width × depth)	60 cm x 26 cm x 48 cm 23 in x 10 in x 18 in
Line voltage	100 - 240 VAC, ±10 %
Line frequency	50 - 60 Hz, ±5 %
Power consumption	700 VA Max
Operating temperature	15 - 30 °C
Non-operating temperature	-40 - 70 °C
Humidity	<95 %, at 40 °C, Non-condensing
Laboratory ventilation	minimum 6 air exchanges/hr for lab air; CO_2 monitor recommended w/ alarm @ 5000 ppm
Exhaust vent capacity	>20 L/min with sustained negative pressure
Operating altitude	up to 2000 m 6500 ft
Non-operating altitude	up to 4600 m 14950 ft
Safety standards	IEC, EN, CSA, UL Installation Category II, Pollution Degree II For Indoor Use only

The Agilent 1260 Infinity SFC Control Module is heavy (approximately 26 kg or 56 lbs). Enlist the aid of a co-worker to share the lifting load in order to avoid possible injury. It should be positioned on a sturdy bench capable of holding the total weight of the Agilent 1260 Infinity SFC Control Module plus the Agilent 1260 Infinity system.

Table 2 Chemical Specifications (G4301A)

Type	Specifications
Inlet CO_2 bulk purity	>99.99 % vapor; >99.999 % liquid
Inlet CO_2 phase	vapor from non-dip-tube high pressure cylinder; liquid from commercial CO_2 delivery system
Inlet CO_2 supply pressure	40 - 70 bar 580 - 1000 psi
Inlet CO_2 temperature	15 - 30 °C
Wash solvent	HPLC grade alcohol
Liquid coolant	30 % propylene glycol in deionized water; proprietary antioxidants; red dye added for safety
Coolant volume	< 280 mL

Table 3 Wetted Materials Specifications (G4301A)

Type	Specifications
High pressure flow path	300 and 400 series stainless steel PEEK, carbon filled PEEK PTFE, PTFE, FEP, CTFE UHMW PE ruby, sapphire, ceramic
Low pressure flow paths [waste, wash pump, leak tray]	316 stainless steel PEEK PTFE, PTFE, FEP, CTFE CPE; LDPE Tygon PVC
Vapor exhaust	Tygon PVC

Performance Specifications

Table 4 Performance Specifications (G4301A)

Type	Specifications
Hydraulic system	Single piston with proprietary motor control
Total hydraulic volume	<5 mL @ pressure <70 bar <25 mL @ pressure up to 400 bar

Table 4 Performance Specifications (G4301A)

Type	Specifications
Chiller system	Thermoelectric cooling with secondary air/liquid cooling circuit
Back Pressure Regulation (BPR) system	Low volume diaphragm type with proprietary drive control; replaceable BPR head assy; No recalibration required after head replacement
Chiller temperature	-20 – 9 °C
Booster pump speed range	0 – 6000 steps/sec average step rate
Booster pump pressure range	100 – 400 bar up to 5 mL/min demand
Pressure pulsation	<2 % amplitude at pump speed >300 steps/sec and outlet pressure >100 bar
BPR thermal range	40 - 70 °C 104 – 158 F
BPR thermal precision	±1 °C
BPR pressure range	100 – 400 bar
Backpressure accuracy	±1 %
Backpressure precision	±0.5 bar (±0.2 bar typical)
Backpressure thermal precision	±1 °C
Control and data evaluation	Agilent ChemStation for LC with SFC Control Module driver; SFC Control Module Diagnostic Program
Analog in pressure monitoring	1 V FS; one input; range set by calibration to host pump
Communications	USB 2.0; APG Remote: ready, start, stop and shut-down signals; relay contact closure [wash pump only]

Agilent 1260 Infinity System

WARNING

Unspecified Conditions

Operating the instrumentation under conditions other than its intended use might result in a potential safety hazard or might damage the instrumentation.

➔ Never operate your instrumentation under conditions other than those specified by the vendor.

G4225A Vacuum Degasser

Table 5 Physical Specifications

Type	Specification	Comments
Weight	5 kg (11 lbs)	
Dimensions (height × width × depth)	80 x 345 × 435 mm (3.1 x 13.5 × 17 inches)	
Line voltage	100 – 240 VAC, ± 10%	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5%	
Power consumption	30 VA / 30 W / 102 BTU	Maximum
Ambient operating temperature	0 – 55 °C (32 – 131 °F)	
Ambient non-operating temperature	-40–70 °C (-4–158 °F)	
Humidity	< 95%, at 25–40 °C (77–104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

G1315C/D Diode Array Detector and MWD G1365C

Table 6 Physical Specifications

Type	Specification	Comments
Weight	11.5 kg (26 lbs)	
Dimensions (height × width × depth)	140 x 345 x 435 mm (5.5 x 13.5 x 17 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	160 VA / 160 W / 546 BTU	Maximum
Ambient operating temperature	0–55 °C (32–131 °F)	
Ambient non-operating temperature	-40 – 70 °C (-4 – 158 °F)	
Humidity	< 95 %, at 25 – 40 °C (77 – 104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

NOTE

ASTM: "Standard Practice for Testing Variable Wavelength Photometric Detectors Used in Liquid Chromatography".

Reference conditions: cell path length 10 mm, time constant 1 s (equal to response time 2 s), flow 1 mL/min LC-grade Methanol, slit width 4 mm.

Linearity measured with caffeine at 265 mm.

Table 7 Performance Specifications

Type	Specification
Wave length range	190 – 950 nm
Wavelenghth accuracy	±1 nm
Linearity range	2.0 AU at 265 nm
Noise, wet	±0.05 mAU under SFC conditions at 254 nm, 20 % MetOH/80 % CO_2 at optimum preconditioning temperature setting (typically between 37 °C and 40 °C for G1315C or G1365C). ±0.007 mAU under LC condition
Drift	0.9 mAU/h
Data rate	80 Hz
Cell pressure limit	400 bar
Cell path length	10 mm
Cell volume	13 µL

G4303A SFC Autosampler

Table 8 Physical Specifications

Type	Specification	Comments
Weight	15.5 kg (34.2 lbs)	
Dimensions (height × width × depth)	200 x 345 x 440 mm (8 x 13.5 x 17 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	300 VA / 200 W / 683 BTU	Maximum
Ambient operating temperature	4–55 °C (41–131 °F)	
Ambient non-operating temperature	-40 – 70 °C (-4 – 158 °F)	
Humidity	< 95 %, at 25 – 40 °C (77 – 104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

Table 9 Performance Specifications

Type	Specification
Number of samples	100 x 2 mL vials, 30 x 6 mL vials
Injection volume	5 µL for full loop injections (5 µL loop as default) larger loops possible (10 µL, 20 µL), partial loop filling for smaller injection volumes
Injection principle	Fixed loop
Injection precision	<0.3 % RSD for 5 µL
Sample temperature control	4 – 40 °C with sample thermostat
Sample carryover	0.05 %

G4302A Binary SFC Pump

Table 10 Physical Specifications

Type	Specification	Comments
Weight	15.5 kg (34 lbs)	
Dimensions (height × width × depth)	180 x 345 x 435 mm (7 x 13.5 x 17 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	220 VA, 74 W / 253 BTU	Maximum
Ambient operating temperature	0–55 °C (32–131 °F)	
Ambient non-operating temperature	-40 – 70 °C (-4 – 158 °F)	
Humidity	< 95 %, at 25 – 40 °C (77 – 104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

NOTE For use with flow rates below 500 µL/min, or for use without damper and mixer, a vacuum degasser is required.

All specification measurements are done with degassed solvents.

Table 11 Performance Specifications

Type	Specification
Number of co-solvents	1, with SSV pump option 3, with external SSV 12
Precision of flow rate	Same as G1312B, ≤0.07 %0.07 RSD or ≤0.02 min SD, whatever is greater based on retention time at constant room temperature
Precision of composition	Same As G1312B, <0.15 % RSD or <0.04 min SD whatever is greater

G1316C Thermostatted column compartment

Table 12 Physical Specifications

Type	Specification	Comments
Weight	11.2 kg (22 lbs)	
Dimensions (height × width × depth)	140 x 345 x 435 mm (5.5 x 13.5 x 17 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	320 VA / 150W / 512 BTU	Maximum
Ambient operating temperature	0–55 °C (32–131 °F)	
Ambient non-operating temperature	-40 – 70 °C (-4 – 158 °F)	
Humidity	< 95 %, at 25 – 40 °C (77 – 104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

Table 13 Performance Specifications

Type	Specification
Column	Up to 300 mm length x 4.6 mm ID (or less)
Column capacity	Up to 3, with additional 2 x TCCs up to 9
Switching valve	Optional (2/6, 2/10, 8/9, 6 column selector)
Temperature range	Ambient -10 °C to 100 °C
Column compartement temperature accuracy	±0.5 °C
Column compartement temperature stability	±0.05 °C
Active solvent pre-heating and post-conditioning	As standard
Automated method development	Optional



3
Installing the G4309A Agilent 1260 Infinity SFC System

Hardware Installation 40
- General Procedures 40
- Installing Agilent 1260 Infinity SFC Control Module (G4301A) 43
- Preparing the HPLC 62

Software Installation 75
- Installing Agilent 1260 Infinity SFC Control Module Driver Software 75

This chapter provides an overview of the installation and setup of the hardware and software

Hardware Installation

General Procedures

Proper use of wrenches

Some of the plumbing connections require a nut to be tightened onto a fitting. There are often two sets of flats next to each other. Attempting to tighten the nut without securing the other part of the fitting with a second wrench can result in loosening yet another connection upstream or downstream. It is best practice to always hold the fitting with one wrench while tightening or loosening another connection.

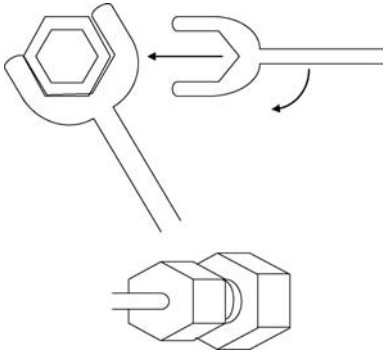


Figure 10 Proper tightening of Fittings

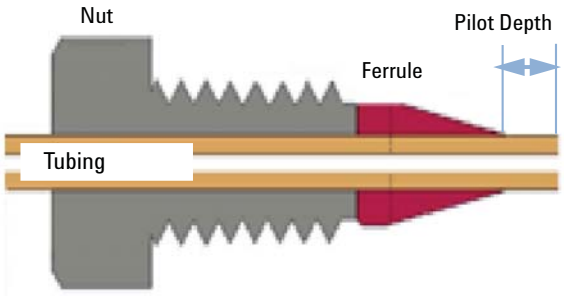
Compression (Swaged Fittings)

The fittings used in the Agilent 1260 Infinity SFC Control Module are Valco. Fittings used in all Agilent modules are Swagelok. Use the appropriate fitting as recommended by the equipment manufacturer. The recommended tightening procedure to install new fittings is to tighten the nut finger tight, then an additional 1/4 to 1/2 turn to seal. In general, previously swaged fittings need only an additional 1/8th turn once finger tight.

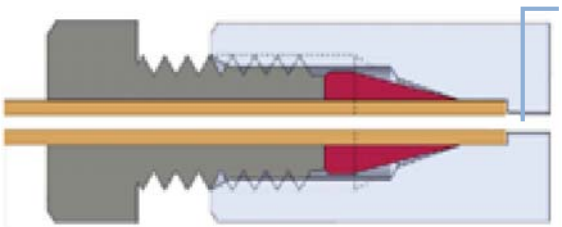
In Supercritical Fluid Chromatography, the fluid has 1/10th the viscosity of water, so this may not be tight enough. All connections should be checked for leaks and tightened further if necessary. Soapy water or Snoop make it easy to find leaks if carbon dioxide is in the fluid. Tiny bubbles appear in the liquid around the fitting.

Each fitting should be individually and carefully installed. The depth of the tube inside the fitting is very important. If the tube pilot (length beyond the ferrule end) is too long, the fitting can leak or, after excessive tightening, bind permanently. If the pilot is too short, a poorly swept volume can be created. This poorly swept volume will create noticeable chromatographic tailing. If the pilot is much too short, the fitting could fail under use. Pilot depths are not always interchangeable between fittings. It is a best practice to swage a tube in the fitting in which it will be used. It is best to provide some light force to hold the tube in the fitting and prevent the tube from exiting while tightening the fitting.

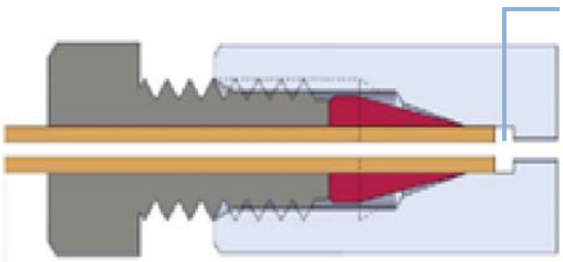
Excessive force can result in breakage of some components, and should obviously be avoided. It may be more expedient to replace the whole fitting if one of the connections fails to seal. You may notice that some of the more expensive components (such as a pressure transducer) have a less expensive fitting mounted to them to act as a sacrificial fitting. Connections should be made to the less expensive component, and repetitive removal and replacement to one of the more expensive fittings should be avoided.



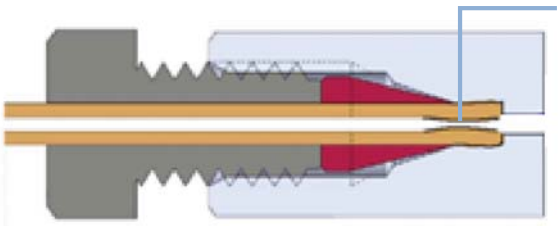
Compression fittings used in the Agilent 1260 Infinity SFC Control Module use a nut and ferrule. As the nut is tightened the ferrule compresses on the tubing creating a leak-tight seal



When correctly installed, there is minimal additional volume. All volume is well swept.



When improperly set, the pilot does not fill the fitting, leading to additional poorly swept volumes.



If over-tightened, the ferrule can compress the tubing and cause sufficient deformation to prevent removal of the fitting.

Figure 11 Compression fittings

Installing Agilent 1260 Infinity SFC Control Module (G4301A)

CAUTION

Early connection may damage the instrument

➔ Do not connect AC power or interconnection cables or gas tubing to the Agilent 1260 Infinity SFC Control Module until these installation procedures direct you to do so.

Preparation

Locate all modules, devices and supporting equipment before continuing. Ensure that the supply tubing can reach a physically secured source of CO_2. Ensure that adequate venting is available and within reach of supplied waste systems.

This document describes a particular order of plumbing the system, with plumbing and electrical connections described last. These operations are performed at the rear of the systems. Depending upon your individual installation, you may wish to perform operations at the rear of the instruments first. This is perfectly acceptable, provided you can maintain access to supply connections to ensure integrity and leak tightness of fittings and connections.

Unpacking the Agilent 1260 Infinity SFC Control Module

Damaged Packaging

When you receive your Agilent 1260 Infinity SFC Control Module (G4301A), inspect the shipping boxes for any signs of damage. If the shipping container or cushioning material is damaged, notify the carrier and save the shipping material for inspection. Save all materials until the contents have been checked for completeness and the instrument has been mechanically and electrically checked.

CAUTION

Signs of damage

➔ If there are signs of damage to the module, please do not attempt to install or use the instrument.

Delivery Checklist

Compare the delivery checklist with the contents of the shipping boxes to ensure completeness of the shipment.

For parts identification see "Parts for Maintenance" on page 119. Please report missing or damaged parts to your local Agilent Technologies representative.

Connecting the Waste system to the SFC control module BPR Outlet

The Agilent 1260 Infinity SFC Control Module has a waste bottle located outside of the cabinet. It can be located anywhere easily accessible and visible within the range of the supplied tubing. The waste bottle serves multiple purposes and collects liquid waste from multiple sources. The primary purpose is to separate the gaseous and liquid waste from the outlet of the BPR (system) in such a manner that the gaseous waste can be appropriately vented outside of the lab environment. The waste bottle has input and output ports located above any collected liquids. The mixed stream enters the waste bottle and the gaseous stream exits from the spout.

NOTE

Proper system operation requires adequate space in the waste bottle to allow gaseous exit. It is the responsibility of the operator to ensure that the waste bottle is empty before beginning operation of the SFC control module, and to monitor and empty the waste bottle as needed during usage. This is not a warning.

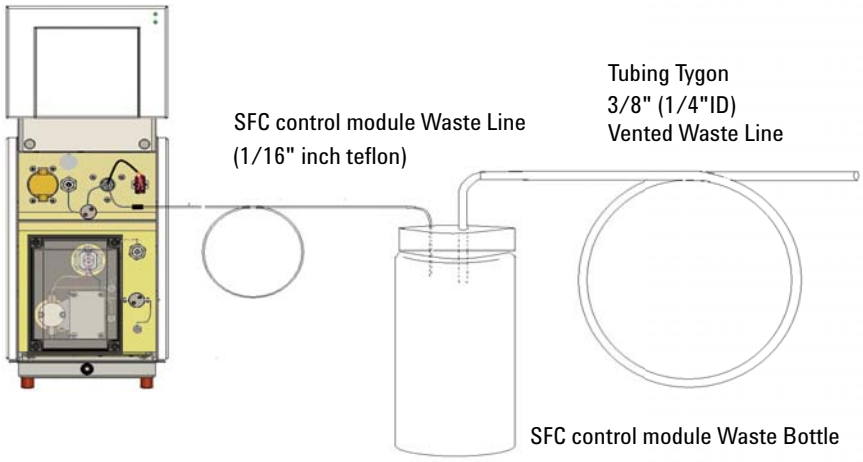


Figure 12 Installing the Waste Container

WARNING

Exposure to toxic substances

➔ The vapor exiting the module may contain several percent organic solvent. The effluent should NEVER be vented directly into an enclosed space occupied by humans because of the potential for long-term exposure to toxic substances.

Locate and assemble the waste bottle and Tygon vent tubing. The vent tubing can be placed over the spout on the top of the waste bottle. Route the Tygon tubing to an appropriate vent. The system must be actively vented.

Locate the SFC control module Waste line. Insert the free end through a hole in the top of the waste bottle cap. Insert the tube half-way into the waste bottle. Connect the fitting end to the outlet union on the BPR. This union uses a 10-32 CPI fitting. Tighten snugly.

Installing the Flowcell

The back pressure regulator exists after any detectors in the HPLC system Thus, the detector flow cells (or splitter in the case on an ELSD or Mass Spectrometer), operate at an elevated pressure relative to HPLC.

Agilent Technologies offers a Diode Array Detector (DAD) flow cell that has been extensively optimized for use in Supercritical Fluid Chromatography (SFC). This cell is pressure-rated and tested to 400 bar. It contains extensive

thermal conditioning not found in standard HPLC flow cells. Agilent Technology's cell is highly recommended for SFC usage.

The flow cell should already be installed in the DAD/MWD. Carefully examine the inlet and outlet ports of the cells to ensure that flow is in the correct direction. In the DAD/MWD, the outlet port is normally located below the inlet port on the connection block.

The inlet port of the DAD/MWD flow cell enters the stationary portion of the handle. This stationary bar acts as an initial thermal conditioning zone. Normally, this port has a male fitting. The outlet port connects directly to the cylindrical portion of the flow cell. Normally, this connection has a female fitting.

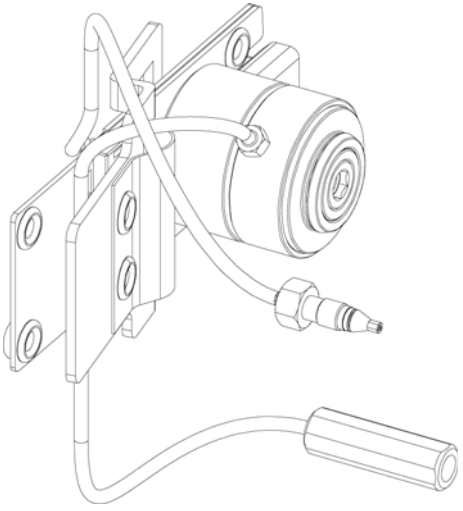


Figure 13 DAD SFC Flowcell

CAUTION

Damage to the flowcell

➔ Verify that the cell installed in the detector is capable of the high pressures used in Supercritical Fluid Chromatography (SFC). Exposing a standard 1260 Infinity flow cell to high pressures will result in leakage or damage to the cell.

Connecting the BPR to the 1260 Infinity stack

Connect the Agilent 1260 Infinity SFC Control Module return transfer tube to the outlet block of the detector. This tube can then be routed out the concave opening in the bottom of the detector behind the detector cover. The return transfer tube should then be routed to the space between the 1260 Infinity stack and the Agilent 1260 Infinity SFC Control Module. Move the tube upward between the units and through the upper tee-slot on the side cover of the Agilent 1260 Infinity SFC Control Module. The return transfer tube can then be fastened in the right port of the tee in the lower center of the BPR drawer.

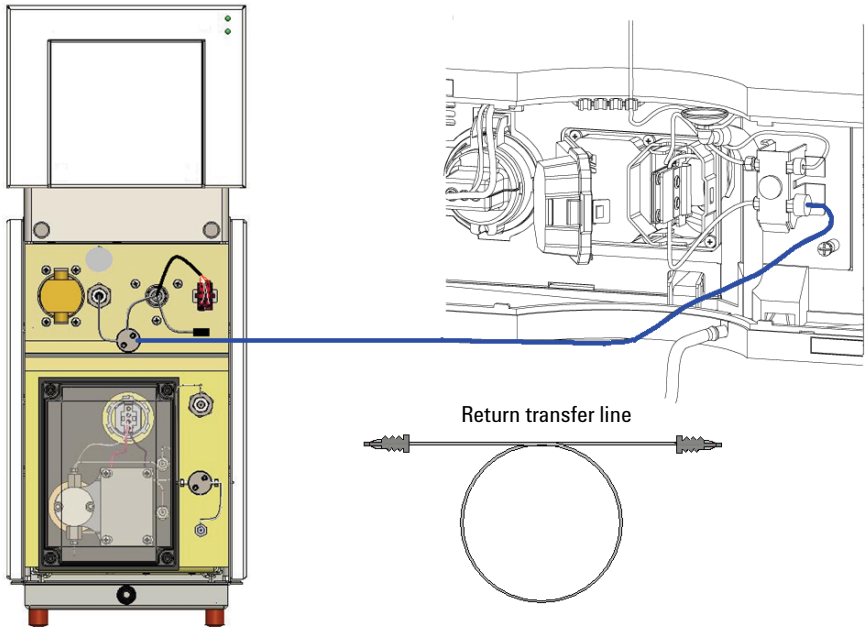


Figure 14 Connecting the SFC control module return Line

Optimized Agilent 1290 Infinity Thermostatted column compartment Plumbing

Supercritical Fluid Chromatography (SFC) is susceptible to increased noise due to poor thermal matching of components within the Agilent 1260 Infinity stack. (The refractive index of carbon dioxide is 50 times more susceptible to temperature changes than water. Consequently, thermal control in SFC is extremely important). The Agilent 1290 Infinity Thermostatted Column Compartment (TCC) contains two thermal conditioning zones that can greatly increase system performance by matching temperatures of the mobile phase to the modules being used.

Each of these zones contains internal transfer lines that can be used to thermally condition the fluid flowing through them. The two zones exist on the left and right side blocks within the TCC.

The block on the right side has a 6 µL internal conditioning volume that is used to precondition the mobile phase before it enters the column. When using 150 mm or shorter columns, place them in the right side of the oven. This zone is used to precondition the fluid to column temperature and provide thermal control of the column.

The left side block is plumbed with the effluent from the column. The purpose of the left side block is to independently match the temperature of the mobile phase to the optimum temperature for the detector. In order to achieve minimum noise it is important to use the optimum conditioning temperature prior to detection (typically between 37 °C and 40 °C for G1315C)

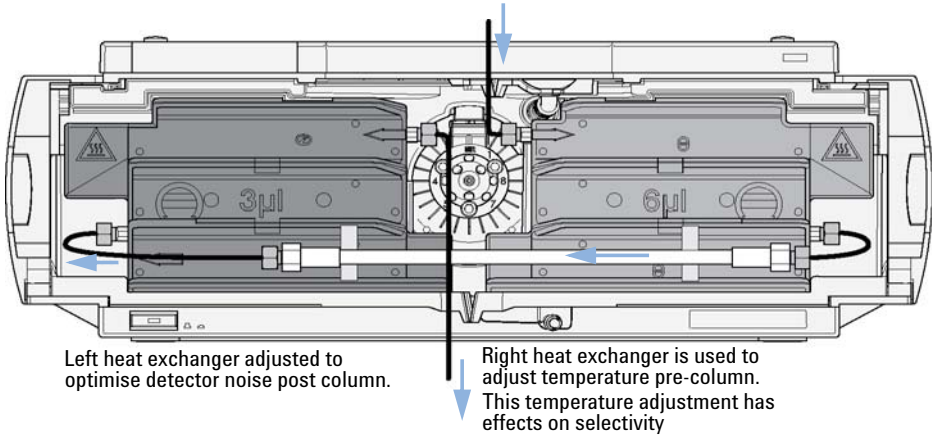


Figure 15 Plumbing the Agilent 1290 Infinity Thermostatted column compartment

Connecting the SFC Autosampler

Agilent Autosamplers need to be converted for use with SFC. If this has not already been done, refer to the installation and upgrade section of the Autosampler compatibility kit for specific instructions.

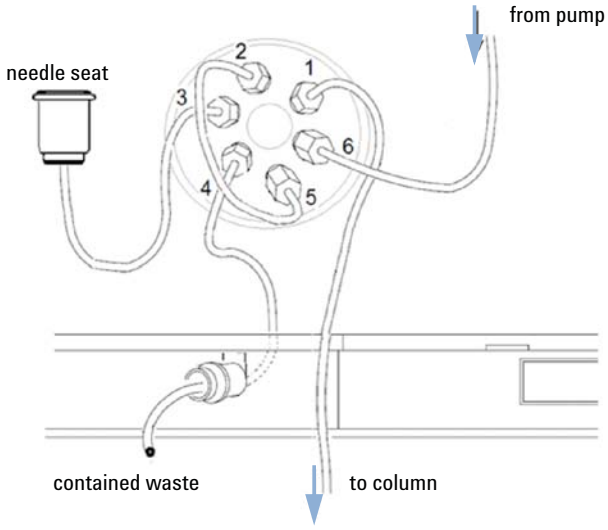


Figure 16 Plumbing the G4203A SFC Autosampler

If the autosampler has been upgraded, the plumbing connections can be made to the injection valve.

In the external loop operation of the autosampler, all connections are made on the injection valve as shown. Port 1 is connected to the inlet of the right side (column pre-heater) thermal conditioning block of the oven.

Port 6 is connected to the output flow from the binary pump.

Connecting the Booster to the Agilent 1260 Infinity system

Locate the stainless steel booster transfer line. Connect one end to the top port of the output tee on the center right side of the booster drawer. Tighten finger tight with an additional 1/8th turn as needed.

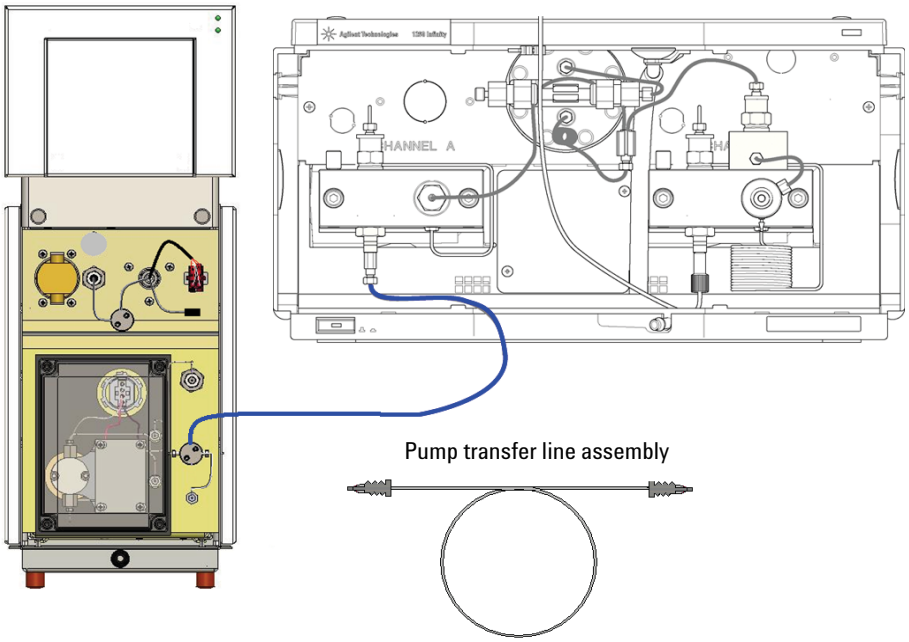


Figure 17 Connecting the Booster outlet line

This tube can be routed through the tee slots on the side of the Agilent 1260 Infinity SFC Control Module. The tube is routed upward between the SFC control module and the Agilent 1260 Infinity stack to the bottom side of the binary pump. The tube is then routed horizontally to the bottom center of the binary pump where it can enter the concave opening behind the cover. The tube should then be routed to the left side of the pump beneath the Channel A (left side). The end of the booster transfer tube can then be installed with the adapter in the passive inlet valve.

Any spare tubing can be located between the SFC control module and HPLC stack.

The injector wash pump

The wash pump is required only with autosamplers normally operated as broken loop autosamplers converted to external loop autosamplers. The injector wash pump requires a source of wash solvent. This wash solvent is used to prime the injector system to ensure proper operation while aspirating sample. If the metering device in the autosampler is not filled with solvent, it can cavitate and yield anything between poor area reproducibility to no peaks (no actual injection). The wash pump also washes the injection system (needle interior, needle seat, injection valve, and sample loop) before and after each injection.

The injector wash pump is connected to a bottle filter (supplied) through the wash pump inlet line. The wash pump inlet line is connected to the lower port on the injector wash pump located on the left side of the BPR drawer. The other end should be connected to the bottle filter. The filter can then be placed in a user-supplied wash solvent bottle.

The wash solvent bottle can be left on the bench or placed in the 1200 Infinity Series solvent cabinet. The wash pump is connected to the metering device (syringe pump) through a check valve intended to prevent siphoning. The operation of the check valve should be verified to ensure it is not leaking, because this can cause a loss of injection precision.

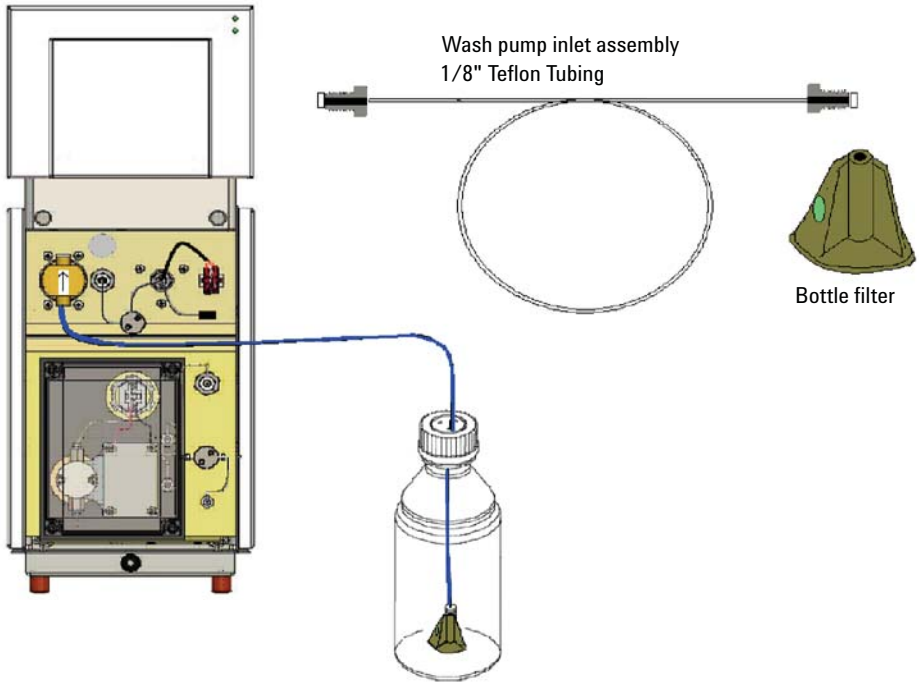


Figure 18 Connecting the wash pump inlet

The outlet of the injector wash pump is connected to the metering device in the Agilent autosampler. To plumb the outlet side of the injector wash pump, locate the was pump transfer line with check valve and install it in the upper port of the injector wash pump. Flow through the wash pump is in the upward direction. The injection wash system will not function if the check valve is improperly installed. An arrow embossed on the check valve body indicates the direction of flow; verify that the check valve is firmly installed.

Connect the wash pump transfer line with the spring loaded check valve. A union is provided on the other side of the of the wash pump transfer line. Follow instructions below for priming the injector wash pump before connecting the union and the autosampler metering device.

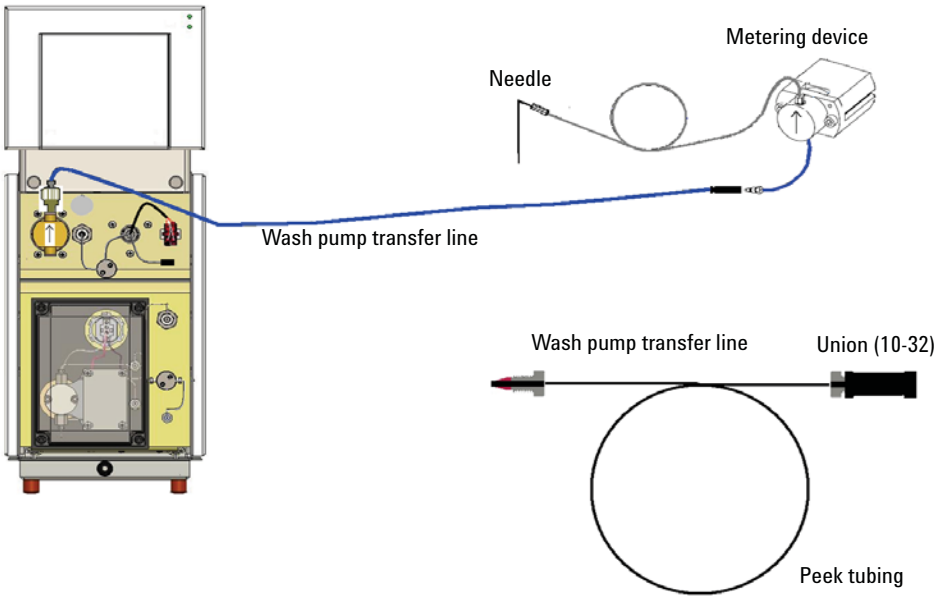


Figure 19 Connecting the Wash Line and Check Valve

Priming the Injector Wash Pump

The wash pump is NOT self priming; it must be filled with wash solvent. See the plumbing scheme, Figure 19 on page 53. There is an extra in-line check valve downstream of the wash pump. This check valve contains a ball pushed into a seat using a spring. The purpose of the spring is to prevent siphoning of the wash solvent from the container, through the injection valve to waste. With the check valve in place, it is easy to prime the injector wash pump without siphoning.

Once the wash pump transfer line and spring loaded check valve are connected to the injector wash pump, you can fill the pump through the injector wash pump transfer line. A syringe and several Luer adapters are included in the ship kit (see Figure 20 on page 54). The syringe can be connected to the wash pump transfer line (once the union is removed) using the Luer adapter. Retracting the syringe pulls solvent through the system, check valves, and tubing. This effectively primes the injector wash pump. After priming, remove the Luer adapter, reinstall the union, and connect to the autosampler metering device.

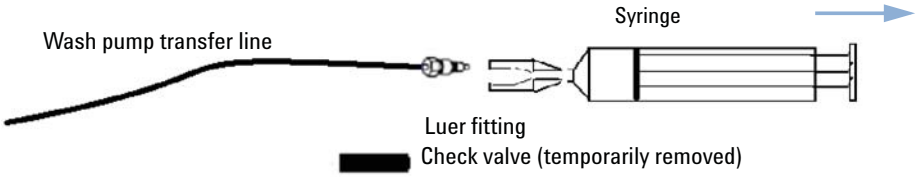


Figure 20 Priming the wash line

Connecting Agilent 1260 Infinity SFC Control Module to a Source of Carbon Dioxide

The Agilent 1260 Infinity SFC Control Module has a 1/8th inch tube inlet connection on the lower left side on the back of the module. This connection is actually part of a very high surface area filter intended to intercept catastrophic levels of particulates. You need to connect this input to a source of carbon dioxide. The most common source of carbon dioxide is liquefied carbon dioxide from a room-temperature cylinder. At room temperature, the pressure in the cylinder could change from a little above 50 bar to just below 70 bar. Unlike most SFCs, Agilent 1260 Infinity SFC Control Module is immune to the variations in flow resulting from cylinder pressure.

WARNING

Creating severe frostbite in a short time

➔ Expanding carbon dioxide can become extremely cold, capable of creating severe frostbite in a short time. Avoid contact with expanding gases. Do not vent substantial quantities into the laboratory.

WARNING

Use the system in a well ventilated area

➔ Carbon dioxide is poisonous at high concentrations and should only be used in well ventilated areas. The system effluent should be vented into a fume hood or to the outside. Evacuate if a large spill occurs. A carbon dioxide sensor/alarm is recommended.

Individual cylinders

WARNING

CO_2 cylinders can be dangerous if handled improperly

➔ Carbon dioxide in cylinders is partially liquefied under high pressure and contains a great deal of energy. If containment is breached (a break in the line or cylinder) the entire contents will vaporize and quickly expand up to 500 times in volume and create very forceful high velocity gas streams. Cylinders must be properly constrained, and proper tubing used, to avoid damage that could generate projectiles.

Any industrial grade of carbon dioxide is acceptable provided it is supplied in a cylinder without a DIP tube. Drawing off the vapor phase leaves non-volatile contaminants behind in the cylinder. Using cylinders with a DIP tube subjects the chromatograph to contaminants soluble in the dense, liquid layer.

Larger tanks are more convenient in that they require to be changed less frequently. Cylinders can contain up to 35 kg of CO_2. Generally, 4.6 mm columns are run at 3 – 5 mL/min, which is approximately 2.5 – 4 g/min of carbon dioxide. This is equivalent to 150 – 250 g/h; 1.2 – 6 kg/day. Thus, a 15 kg cylinder should last 2.2 to 11 days; a 25 kg cylinder would last 4 to 19 days; and a 35 kg cylinder could last 5.3 to 27 days - all depending on use (3 – 5 mL/min; 8 – 24 h/day). For individual users, particularly new users, the use of cylinders is perfectly acceptable. Larger groups should consider installing a gas delivery system and a bulk storage tank.

Locate the cylinder as close to the instrument as possible. In the past, the cylinders were stored at much higher temperature than the lab temperature, which resulted in vaporization in the supply line coming into the lab. Most SFC pumps cannot condense this vapor and therefore, cannot deliver CO_2. Agilent 1260 Infinity SFC Control Module has a very powerful condenser designed to accept vapor phase CO_2. Nevertheless, it is always advisable to not stress any equipment.

Facilities and safety personnel often wish to store and mount the cylinders outside the lab — sometimes quite far from the intended location of the instrument. They should recognize that the transfer lines can hold large volumes, equivalent to a large fraction of a cylinder, particularly if tubing with large ID is used. Shut-off valves at both ends of a transfer line are not recommended, unless one or both has a pressure relief valve or burst disk.

Be sure the cylinder is properly constrained and cannot tip over. Suitable chains or cylinder straps are required.

Cylinders in the USA and Canada use a CGA 320 cylinder adapter. One is included in the module USA ship kit, along with a 1/4 inch FNPT to 1/8 inch compression fitting, and 6 feet of 1/8 inch OD stainless steel tubing. The filter fitting sticking out the back of the Agilent 1260 Infinity SFC Control Module contains 1/8 inch nut and ferrule(s), which could be used with the supplied 6 feet tube, or a longer, user-supplied tube, to connect a cylinder to the SFC control module.

There are at least four different European standards for the connection of carbon dioxide cylinders. They do not appear to change at national borders (some users in one country have different supply connections from other users in the same country). Agilent Technologies recommends that customers in Europe contact their gas supply companies and ask them how to mate the cylinders they supply with an American 1/8 OD supply line. This should require a cylinder connector and a reducing union down to a 1/8 inch compression fitting.

WARNING

Leaks will not be sensed or protected

➔ Carefully check supply cylinders and inlet fittings for leaks. Any leaks present in the supply line and inlet fitting will not be sensed or protected by safety features in the module.

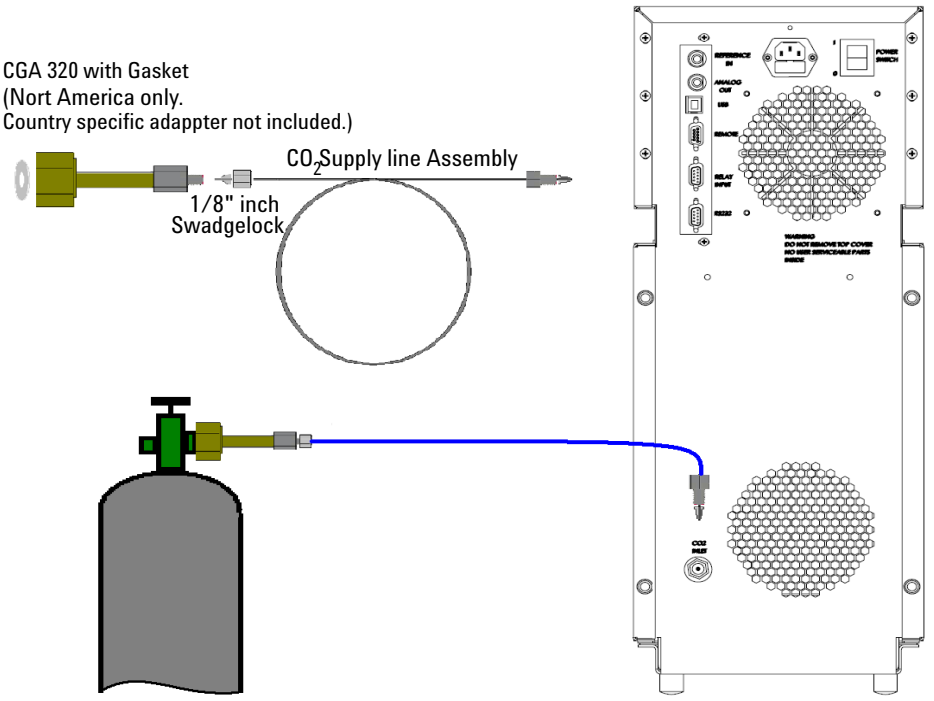


Figure 21 Figure 3.25

0	Power off
I	Power on

WARNING

Improper plumbing can cause leaks

➔ It is imperative to use two wrenches to install the supply fitting in the bulkhead filter (entry connection) on the back of the module. Turning or twisting the bulkhead supply filter could cause failures or leaks in tubing within the module.

➔ All supply fittings need to be thoroughly checked for leaks. Any leaks in the supply fittings can vent the CO_2 supply.

CAUTION

Overtightening the fitting could damage the filter

➔ Use two wrenches when installing the CO_2 supply line to the bulkhead filter (entry connection) on the back of the module. Although the filter is captured and should not rotate, there is a remote possibility that fittings inside the module could be loosened by severe stress on the nut on the supply line. Leakage inside the cabinet requires service by trained personnel.

Gas Delivery Systems (GDS)

Anyone performing semi-prep SFC has probably been convinced that operation without a GDS is problematic. Thus, many laboratories are now plumbed with carbon dioxide boosted to 70 – 80 bar outlet pressure. Even though an analytical system does not need such a GDS, it is perfectly adapted to its use.

The Agilent 1260 Infinity SFC Control Module has an inlet safety shut-off valve rated to 1500 psi (>100 bar). While this is rating is well above the outlet pressure of any typical GDS, past experience suggests that it is wise to allow for some extra margin. Agilent Technologies suggests setting the local output of any GDS between 60 bar and 70 bar, through local outlet pressure regulators.

Under these conditions, the GDS will almost always provide liquid carbon dioxide to the chromatograph. Unlike some earlier systems, the Agilent 1260 Infinity SFC Control Module easily condenses any fluid that is present as a vapor, and prevent pump cavitations.

Cable connections to the HPLC

WARNING

Personal injury

➔ Ensure that the AC power cord is NOT yet connected to the instrument.

Cable connections to the Agilent 1260 Infinity SFC Control Module are dependent upon the installed configuration. The primary decision to be made is whether or not an Agilent autosampler is present. When an Agilent 1260 Infinity SFC Autosampler is present, the Agilent 1260 Infinity SFC Control Module remote line and relay contacts lines are connected to the autosampler.

The Binary pump analog output signal should be connected to the Agilent 1260 Infinity SFC Control Module **Reference in** terminal.

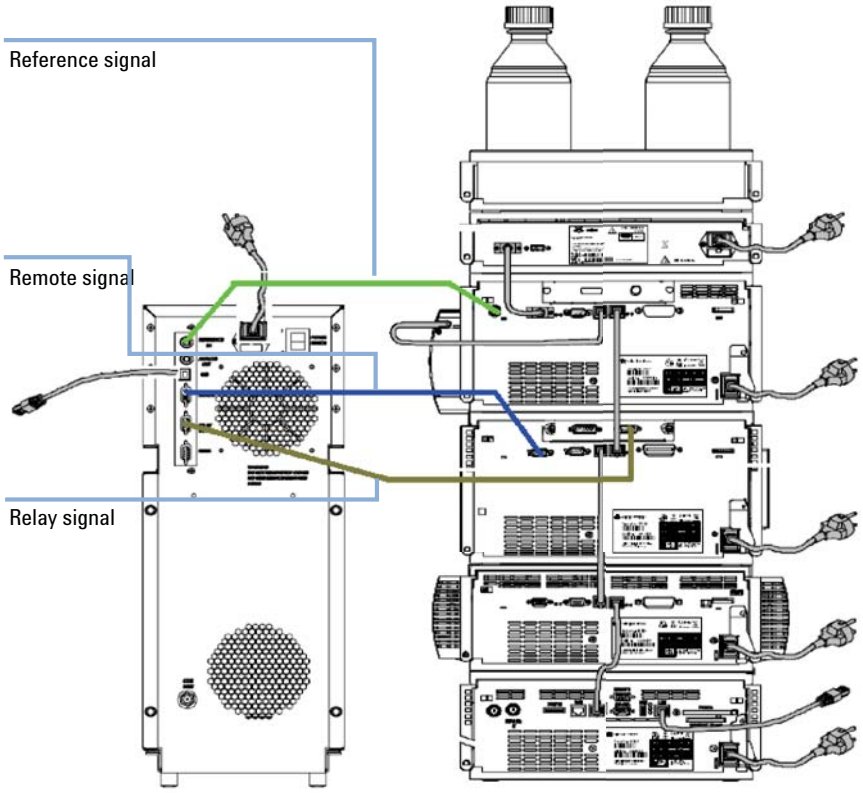


Figure 22 Reference, Remote and Relay Signal Connections

0	Power off
I	Power on

Lastly, a cable should be connected to the Agilent 1260 Infinity SFC Control Module USB port. This can be run to any convenient, compatible USB port on the PC.

CAN cabling between the Agilent 1260 Infinity modules is unchanged from the normal, recommended means of interconnecting HPLC devices.

The Agilent 1260 infinity stack requires a LAN connection to the PC. This LAN connection requires all the normal HPLC/ChemStation properties such as BOOTP, Firewall, and IP Address settings. These remain unchanged in an Agilent 1260 Infinity SFC Control Module installation.

Connecting and operating multiple instruments on a single PC is not supported. Both the Agilent 1260 Infinity SFC Control Module USB and Agilent 1260 Infinity LAN connections must be made on the PC on which ChemStation is installed and which is used for instrument control.

Connecting the Leak Tray Waste Line

The Agilent 1260 Infinity SFC Control Module contains a leak tray on the bottom of the instrument to collect and sense any liquid spills that may occur in the Agilent 1260 Infinity SFC Control Module cabinet. In the bottom of this leak tray is an active sensor that continuously monitors for the presence of liquid.

The drip tray contains an overflow drain to divert any large amounts of collected liquid to an external collection container. The overflow tube incorporates a simple push-to-connect fitting. It is connected to the port on the bottom center on the front of the instrument. Pushing on the outside ring of the port allows this line to be removed.

Since this liquid may be organic solvents, you should supply an appropriate collection container.

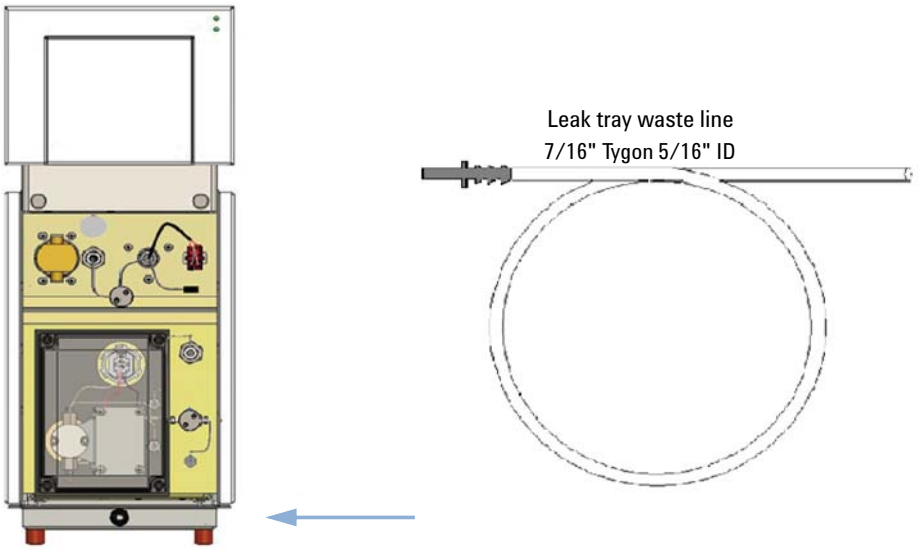


Figure 23 Leak Tray Waste Line

Preparing the HPLC

Preferred Stack Arrangement

To minimize delays and broadening caused by excessive tube lengths, we recommend the following stack layout. The Agilent 1260 Infinity SFC Control Module needs to be immediately adjacent to the LC stack, but can be placed on either side. In the preferred arrangement, the degasser and solvent tray are located on the top of the stack. These feed into the binary pump, and the SFC control module injection wash pump. The binary pump is located below the degasser and directly above the autosampler. The thermostatted column compartment is located below the autosampler, with the detector at the bottom of the stack.

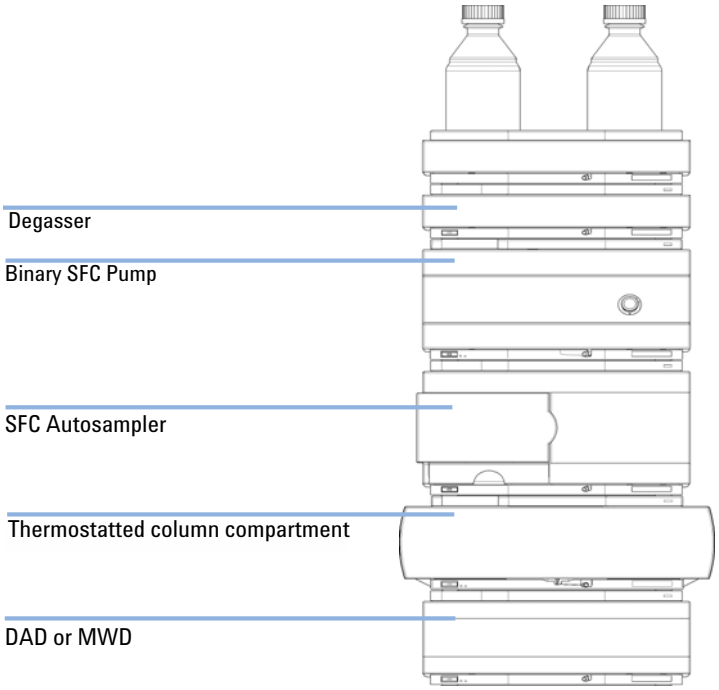


Figure 24 Preferred order of Agilent HPLC Components

It is recommended to plumb the system with small diameter tubing. For general use 0.17 mm or 0.007 inch tubing should be used. In more demanding applications where pressure drops are not excessive, 0.12 mm or 0.005 inch tubing can be selectively used. Use ferrules and tubing connections as recommended by the equipment manufacturer.

All four degasser channels will be available for solvent usage. It is highly recommended that one channel be reserved for neat methanol (no additives). Reserving this channel will minimize the time needed for flushing the degasser as solvents are changed. Failure to adequately flush the degasser when switching solvents will greatly impact sensitivity.

Agilent 1260 Infinity SFC Binary Pump

It is highly recommended that you familiarize yourself with standard maintenance functions and terminology used in the binary pump. This information is available in the Agilent Binary pump reference manual and user guide.

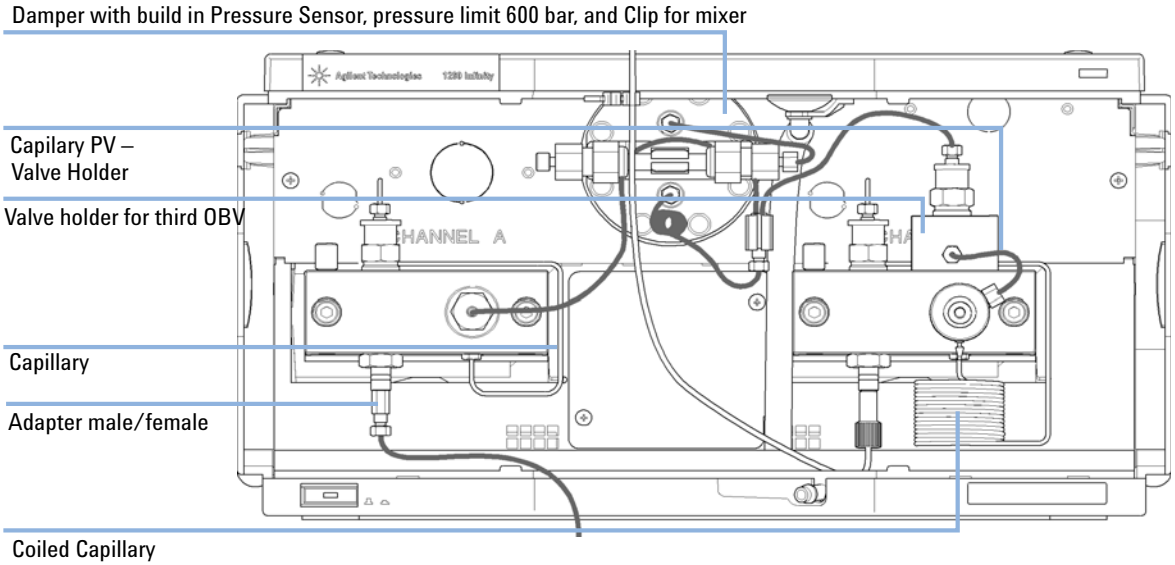


Figure 25 Diagram of the Agilent Binary Pump

The G4303A SFC Pump is a binary high pressure mixing pump, optimized for the usage in a SFC System. The Purge Valve is mounted on the right side for purging the Modifier Channel (Channel B) only. An additional third Outlet

Ball Valve (OBV) is installed with a Valve Holder on Pumphead B, to allow the Channel B to be flushed, even if CO_2 tank pressure is supplied to the System. On the Passive Inlet valve of the CO_2 Channel (Channel A) is an Adapter to allow a Swagelok fitting to be connected to the valve. The damper includes the pump pressure sensor and allows a pressure up to 600 bar. Different set of piston seals are installed in the two pump heads. Normal phase seals (yellow PE) in pump head channel A, standard seals PTFE, carbon filled in pump head channel AB.

WARNING

Toxic, flammable and hazardous solvents, samples and reagents

The handling of solvents, samples and reagents can hold health and safety risks.

- ➔ When working with these substances observe appropriate safety procedures (for example by wearing goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet supplied by the vendor, and follow good laboratory practice.
- ➔ The volume of substances should be reduced to the minimum required for the analysis.
- ➔ Do not operate the instrument in an explosive atmosphere.

The BNC coaxial cable can be used to connect the Analog Output on the back panel of the Binary pump to the Agilent 1260 Infinity SFC Control Module Reference In terminal/connector on the back of the SFC control module. Be aware that the output of similar appearance on the DAD and VWD detectors should NOT be used.

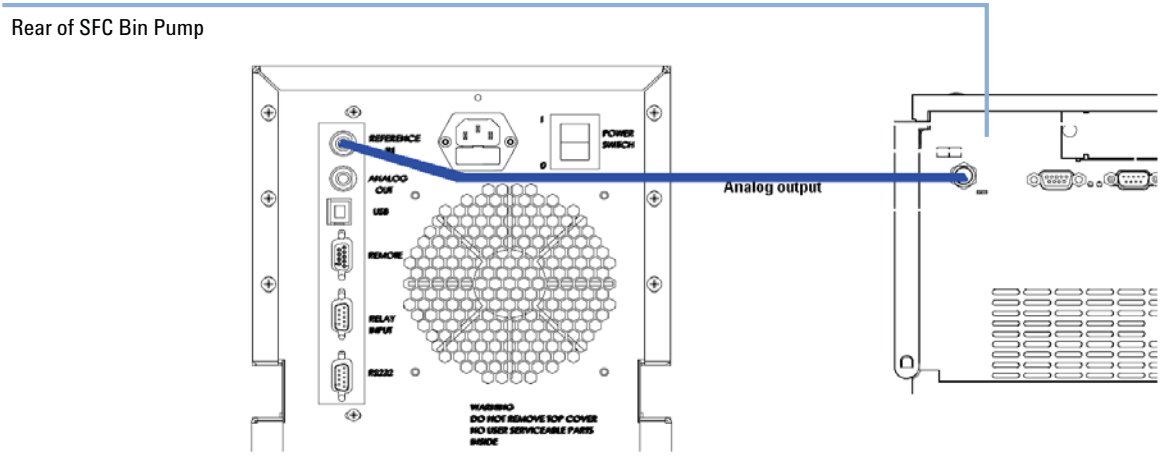


Figure 26 Connecting the Analog Reference Signal

0	Power off
I	Power on

Modifications to the Agilent Autosampler

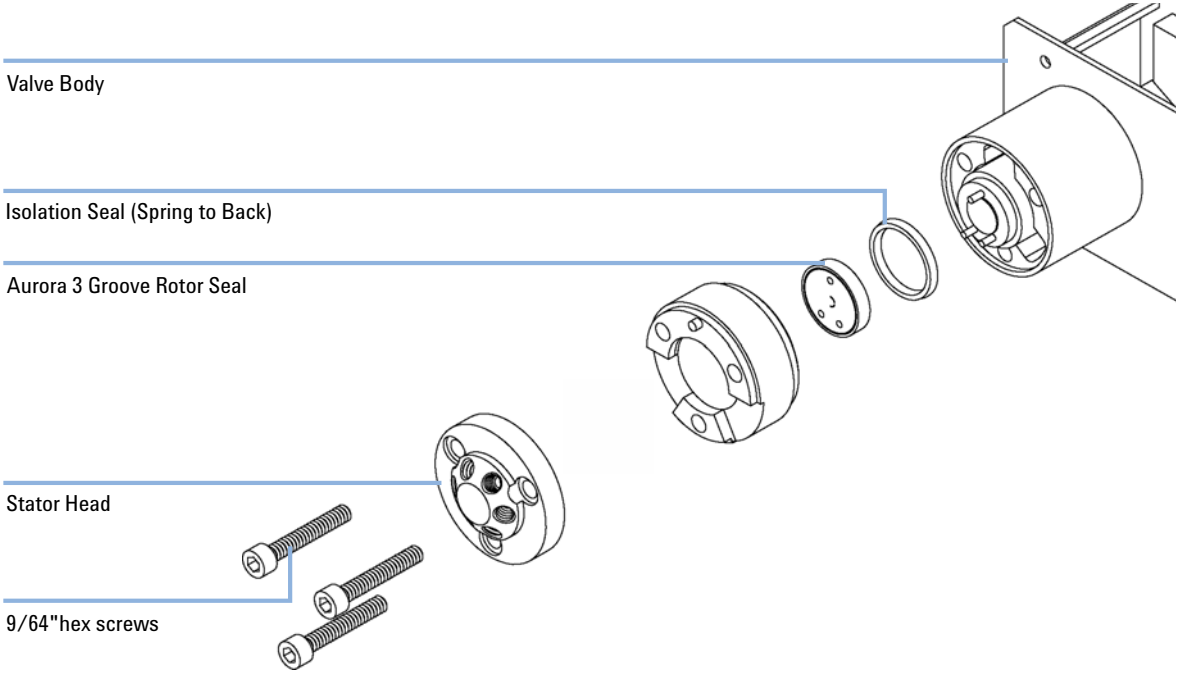


Figure 27 Exploded View of the Autosampler Injection Valve

NOTE Replacing a rotor seal is well documented in Agilent manuals, and service videos. Please refer to these guides for further assistance.

1 The Autosampler should be plumbed in accordance with Figure 28 on page 67.

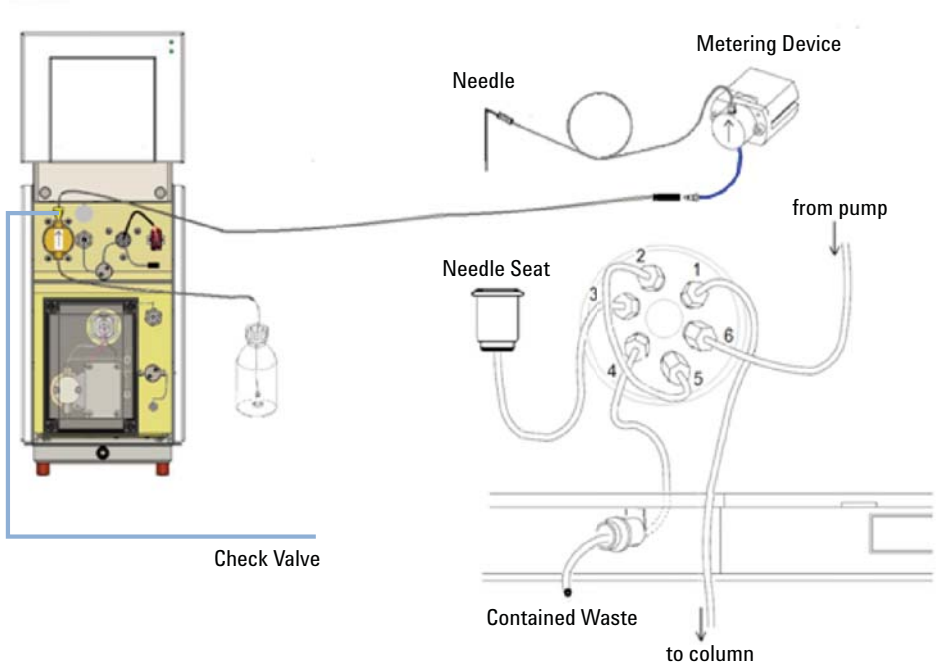


Figure 28 Autosampler Fluidic connections

2 Connect one end of the wash pump transfer line with the check valve to the outlet port of the injection wash pump.

3 Before connecting the union end of the wash pump transfer line, use a syringe and Luer Adapter to prime (pull sample through) the injection wash pump

4 The existing line (blue capillary) from the inlet of the Agilent 1260 Infinity SFC Autosampler metering device can be plumbed into the union on the wash pump transfer line

Positioning the waste line

In normal operation, the sample loop is switched from mainpass i(n the high pressure flow stream) to bypass (out of the flow stream and connected to the seat capillary and waste). As the valve switches, the fluid in the loop expands and rushes out of the loop through the waste line. If the waste line is positioned to empty into a liquid reservoir such as a WPA flush port, the exiting fluid from the sample loop could splash the flush port fluid across the ALS. The preferred positioning of the waste line is through the leak vent. In this position, the expanding fluid exits harmlessly into the leak tubing.

Alternatively, a waste line can be fabricated that empties into the Agilent 1260 Infinity SFC Control Module gas liquid separator (waste bottle).

Under no circumstances should the waste line be allowed to be unconstrained. During operation of the injection wash pump, this is the final exit of the wash fluid.

Installing the Agilent BCD Board

The BCD Interface card (Interface board (BCD) with external contacts and BCD outputs (G1351-68701)) and relay cable from the shipping kit are used to control the wash pump.

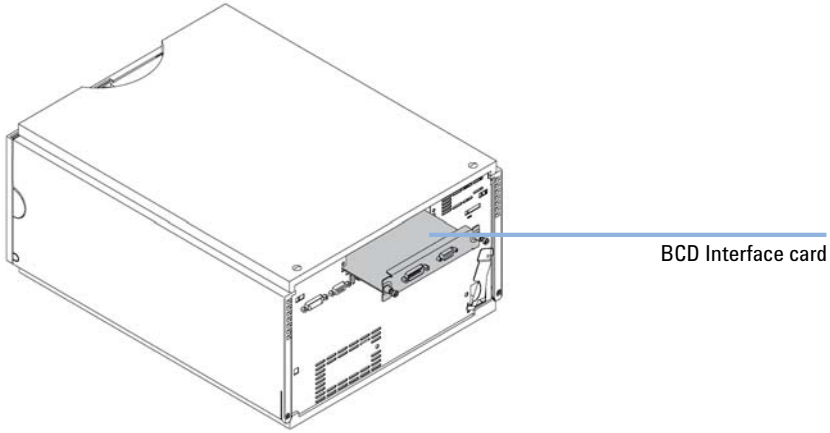


Figure 29 BCD Interface Card

Agilent HPLC autosamplers have a small compartment located on the back panel near the top, with a cover held in place by two captured knurled nuts.

Loosen the nuts and remove the cover, taking care to retain it for future use. Take the BCD Interface card from the shipping kit, taking care to prevent damage from static electricity, solvent, etc.

CAUTION

Electronic boards are sensitive to electrostatic discharge (ESD) and should be handled with care so as not to damage them. Touching electronic boards and components can cause electrostatic discharge.

ESD can damage electronic boards and components.

➔ Be sure to hold the board by the edges and do not touch the electrical components. Always use an ESD protection (for example, an ESD wrist strap) when handling electronic boards and components.

1 Carefully slide the board into the slot. When it is fully in place, gently push the bezel until the connectors engage. Tighten the captive knurled nuts.

2 With the BCD card in place, connect the 15-pin relay contacts connector to the relay input connector on the back of the SFC control module.

3 Connect the 9-pin Remote connector on the back left of the autosampler to the 9-pin Remote connector on the back of the SFC control module.

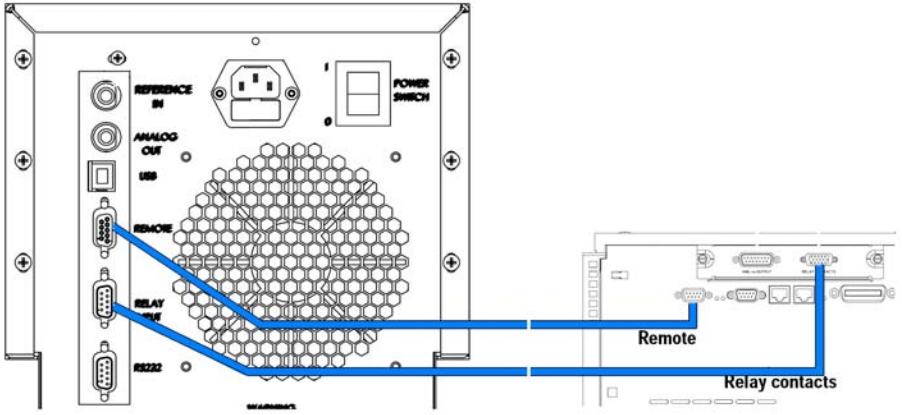


Figure 30 Autosampler Signal Cable Connections

0	Power off
I	Power on

Installing an Injection Program in the Method

The injector program is delivered as part of the default setup, but you can enter it manually, if necessary.

Table 14 Agilent 1260 Infinity SFC Autosampler Injector Program

Injection	
Injection Volume	15.0 µL
Injection Mode	Injector Program
Time	
Stoptime	No Limit
Posttime	Off
High Throughput	
Autom. Delay Vol. Reduction	Off
Overlapped Injection	disabled
Minimized Carry Over	Off
Needle Wash	
Wash Mode	Wash in Flushport
Wash Time	1.0 s
Wash Location	Vial 10
Repeat	1 times
Injector Cleaning	
Injection Valve Cleaning / Valve Switching	
Time 1 Bypass	off
Time 2 Mainpass/Bypass	off
Time 3 Mainpass/Bypass	off
Time 4 Mainpass/Bypass	off
Valve Movements	1
Auxiliary	
Draw Speed	100 µL/min
Eject Speed	100 µL/min
Draw Position	0.0 mm

Table 14 Agilent 1260 Infinity SFC Autosampler Injector Program

Equilibration Time	2.0 s
Sample Flush-Out Factor	5.0 times Injection Volume
Vial/Well Bottom Sensing	No
Store Temperature	No
Injector program table	
Row	Action
1	NEEDLE down
2	CONTACT B CLOSED
3	WAIT 0.10 min
4	CONTACT B OPEN
5	VALVE bypass
6	DRAW 1.5 µL from air, def. speed
7	DRAW def. amount from sample, def. speed, def. offset
8	DRAW 5 µL from air, def. speed
9	NEEDLE wash as method
10	EJECT max. amount into seat, def. speed
11	VALVE mainpass + start pulse
12	CONTACT B CLOSED
13	WAIT 0.10 min
14	CONTACT B OPEN
Agilent Contacts Option	
Contact 1	Open
Contact 2	Open
Contact 3	Open
Contact 4	Open

NOTE Using the Agilent SFC Autosampler as a fixed loop to (external) loop autosampler has several minor consequences that need to be understood to achieve adequate performance.

Dead Volume-Partial Loop Injections

Plumbed as a loop injector, the Agilent 1260 Infinity SFC Autosampler has a dead volume between the high pressure needle seat and the groove on the rotor of the injection valve. Two different diameter tubes are used to make this connection. Thus, the actual dead volume can have several different values, depending on the ID of the tubing that is used. This dead volume can be several micro liters or more. When attempting to make low-volume injections, it may be possible that no sample enters the loop unless precautions are taken. Use an air bubble on each side of the sample, insert a plug of modifier or other solvent behind the sample, or any other method to ensure that the sample reaches the loop.

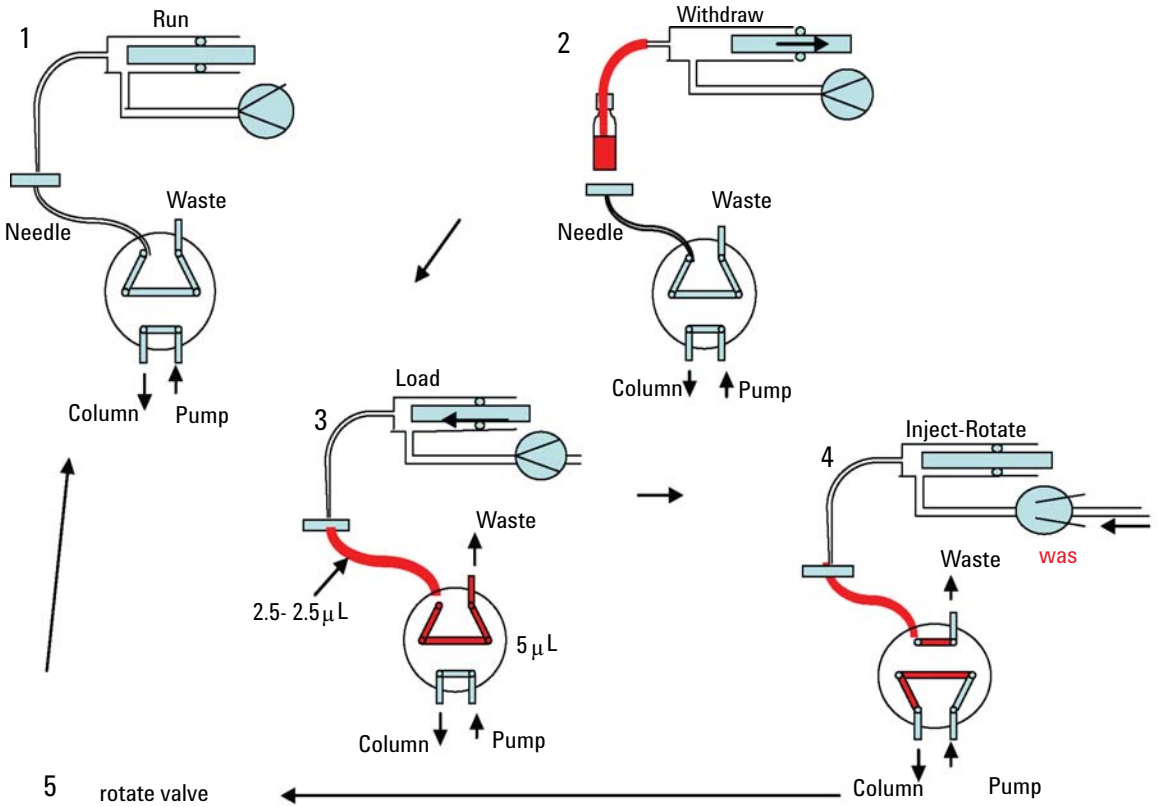


Figure 31 Full Loop Injection

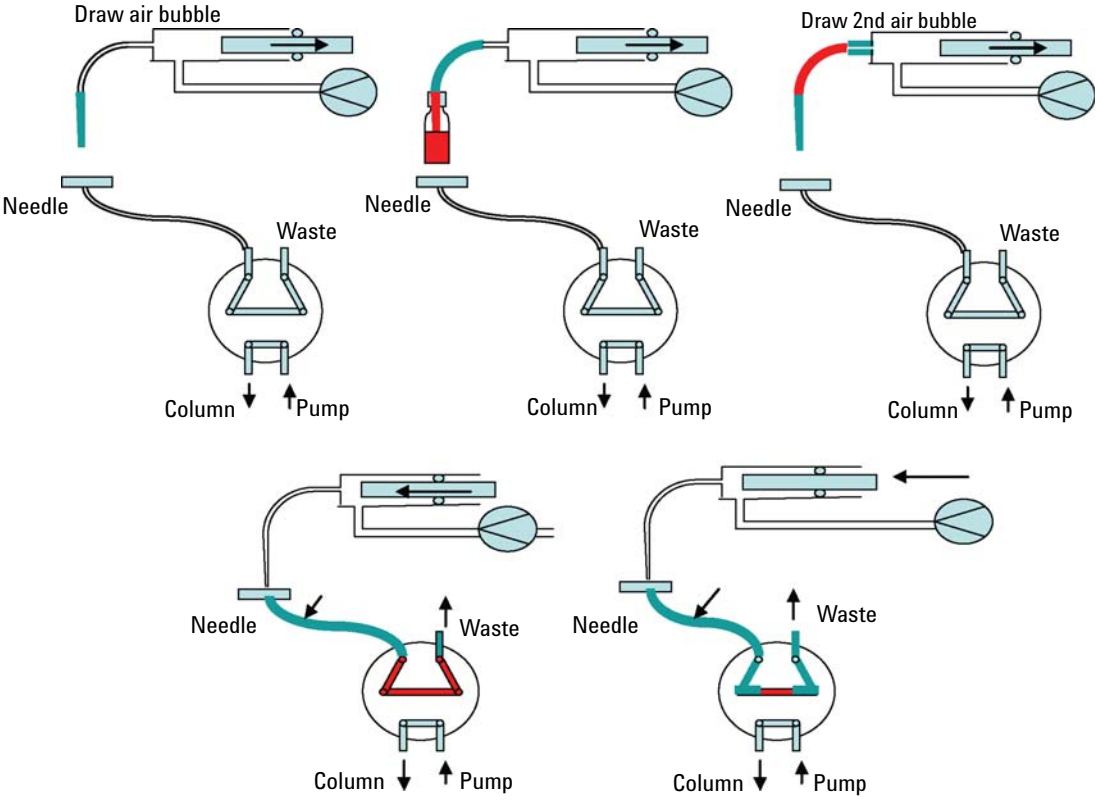


Figure 32 Partial Loop Injection

Some General Injection Rules

20 µL loops are commonly used in HPLC. In SFC, the sample is often dissolved in a solvent that is stronger than the mobile phase. Injecting large volumes, such as 20 µL, of such a solvent will cause peak distortion.

As a rule of thumb, no more than approximately 5 µL of a polar solvent such as methanol should be injected onto a 4.6 mm ID column, as shown in Figure 33 on page 74.

This should NOT have a major impact on area reproducibility but should destroy efficiency as indicated in Figure 33 on page 74.

Use Small Loops

The loop should not be much larger than the maximum desired injection (or =<5 µL). Bear in mind that the loop is washed with a strong solvent, and is usually filled with that solvent. The sample displaces some or all of this solvent.

If the loop is too large, too much strong solvent is injected, regardless of whether the loop is filled with sample or wash solvent. Peaks will be distorted and efficiency will degrade.

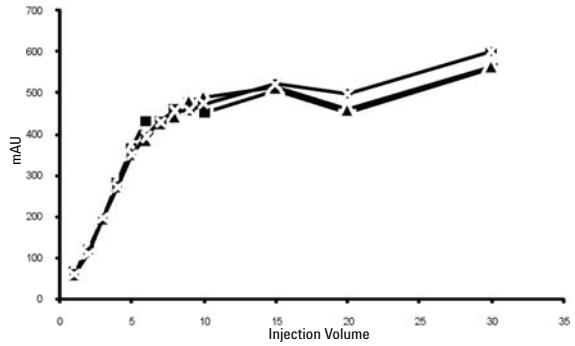


Figure 33 Plot showing loss of efficiency with large injection volume

If a large loop is used, air bubbles can be used to displace the wash solvent.

Less Polar Sample Solvents Help

Replacing the sample solvent with a much less polar solvent can allow injection of much larger volumes. However, the new solvent must be significantly less polar than the modifier used. Replacing methanol with ethanol or even isopropanol has minimal effect. Solvents such as chloroform or methylene chloride tend to cause significant focusing (NO broadening), but since they are chlorinated should probably be avoided.

Software Installation

The software installation consists of the following steps:

1 Install Agilent ChemStation, for more details see *Agilent ChemStation for LC and CE Systems Installation Manual*, Chapter *Installing the Agilent ChemStation, Initial Installation*.

2 Install SFC USB driver.

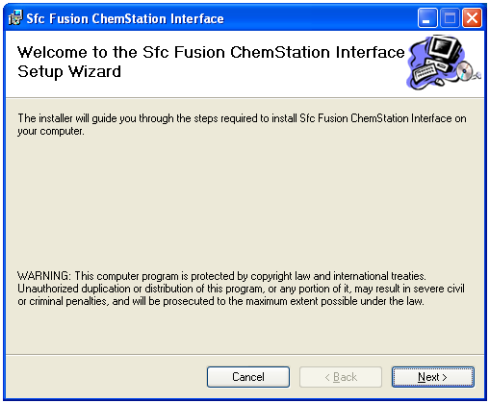
3 Install the Driver Software, see "Installing Agilent 1260 Infinity SFC Control Module Driver Software" on page 75

Installing Agilent 1260 Infinity SFC Control Module Driver Software

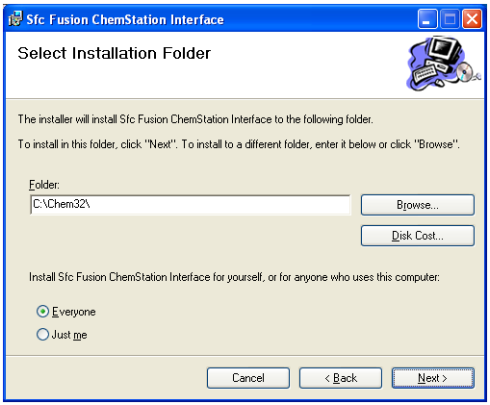
The driver is installed as an add-on product for ChemStation.

The following screenshots show the installation steps. It is important to select the ChemStation installation directory as destination path!

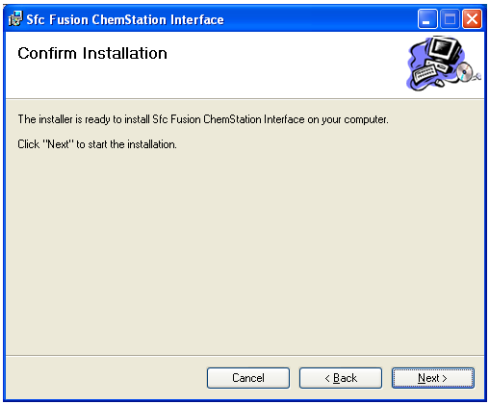
1 Start the **Sfc Fusion ChemStation Interface Setup Wizard**



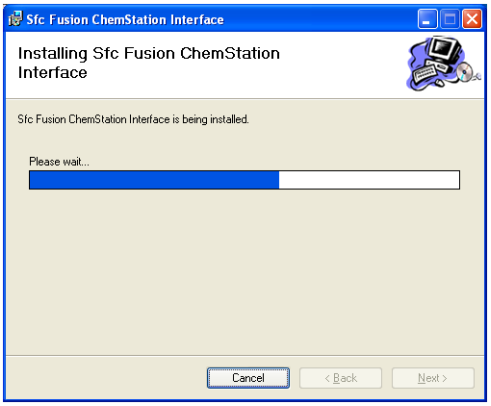
2 Select installation folder.



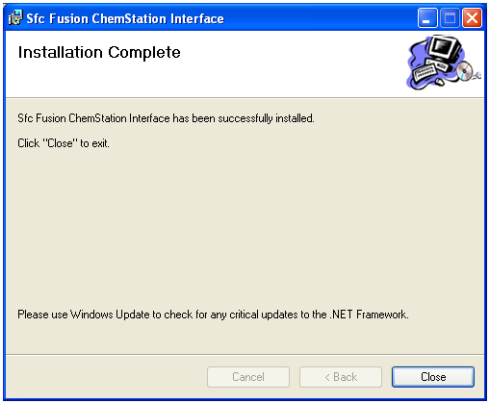
3 Confirm installation.



4 Install the **Sfc Fusin ChemStation Interface**.



5 Complete the installation.





4
Configuring the System

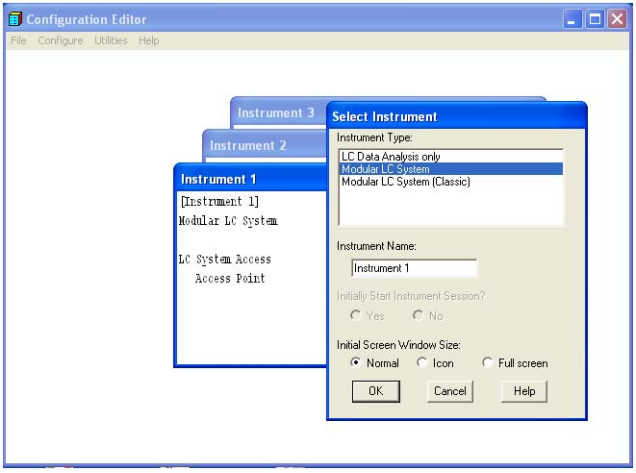
Configuring SFC interface in ChemStation 80
Setting up the Method 84
Status 86
Control 87
Testing the System 88

How to configure the Agilent 1260 Infinity SFC Analytical system and Agilent 1260 Infinity SFC Control Module in ChemStation.

Configuring SFC interface in ChemStation

NOTE The Agilent 1260 Infinity SFC Control Module ChemStation interface works with the ChemStation **Modular LC System**, not with **Modular LC System (Classic)**. The **Modular LC System (Classic)** instrument does not support RC.Net drivers, which are necessary for the LC modules.

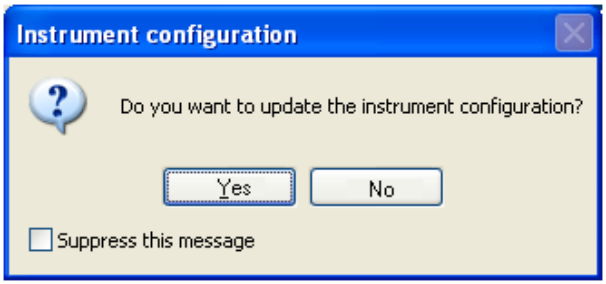
1 Select **Modular LC System** as instrument type.



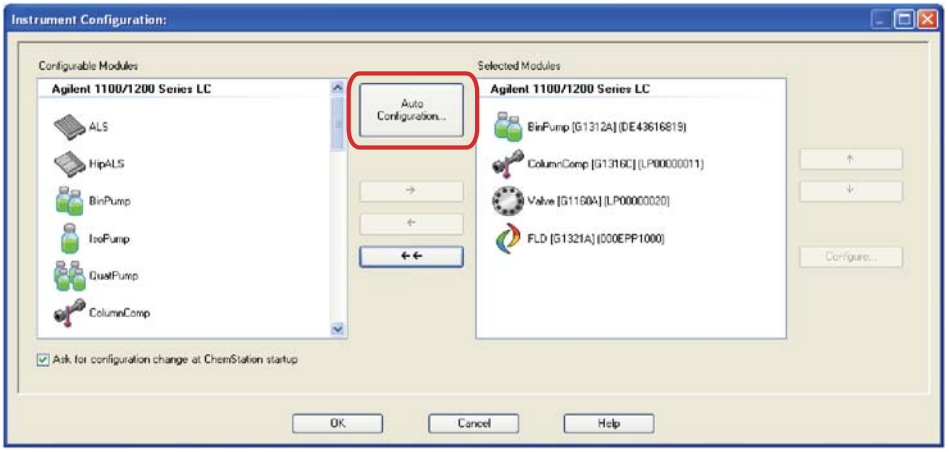
2 Start the Agilent ChemStation.

3 Configure the devices in the Agilent ChemStation, see *Agilent ChemStation for LC and CE Systems Installation Manual*, Chapter *Configuring the Instruments, Configuring a Modular 3D LC System or Modular LC System*.

4 Depending on the current configuration, Agilent ChemStation startup phase asks if the configuration shall be modified.



5 In the **Instrument Configuration** editor **Auto Configuration** sets up the Agilent system as usual.

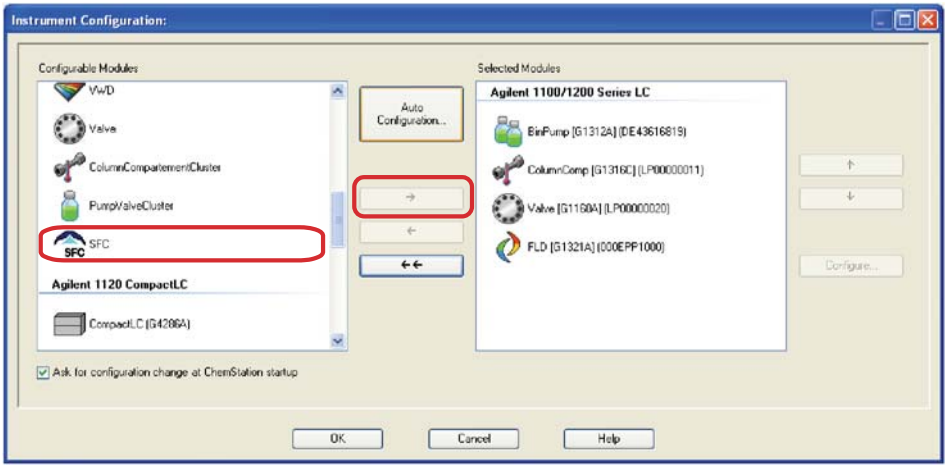


CAUTION

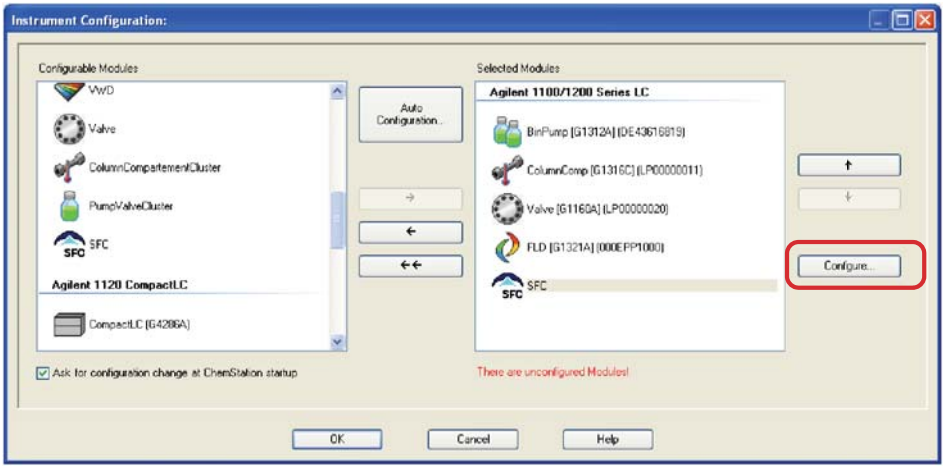
Conflict with SFC macro implementation

➔ It is important that the SFC Control Module is listed below the Agilent pump; otherwise, the SFC will become pump 1 of the system, which conflicts with the current SFC macro implementation. The SFC macro always checks the first pump of the system for error or standby.

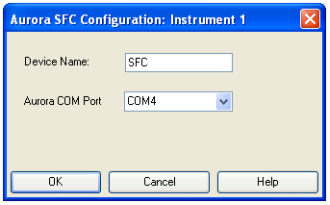
6 To set up the SFC module, scroll down the **Configurable Modules** slider until you see the SFC icon. Select it with a double-click or right arrow to move it to the **Selected Modules** panel.



7 Select **Configure** to open the SFC configuration dialog box.

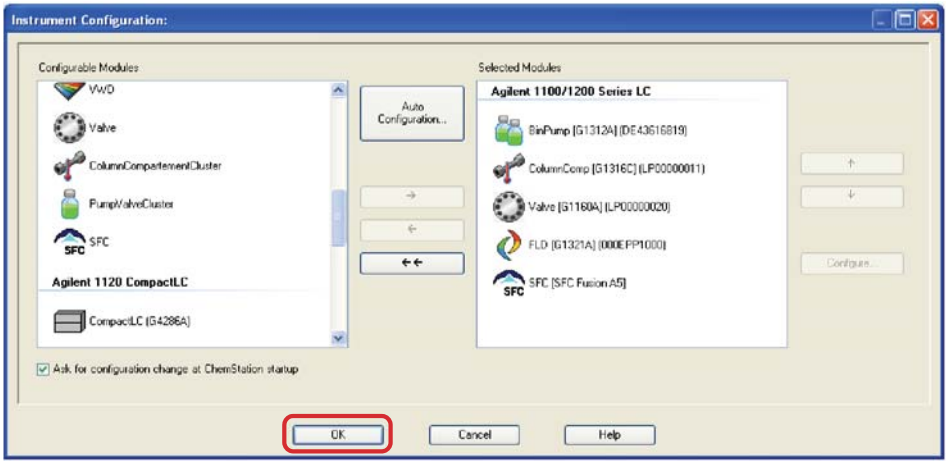


8 In the **Aurora SFC Configuration**, enter the device name or use the default to identify the module within Agilent ChemStation. Select a COM Port in the **Aurora COM Port** field.



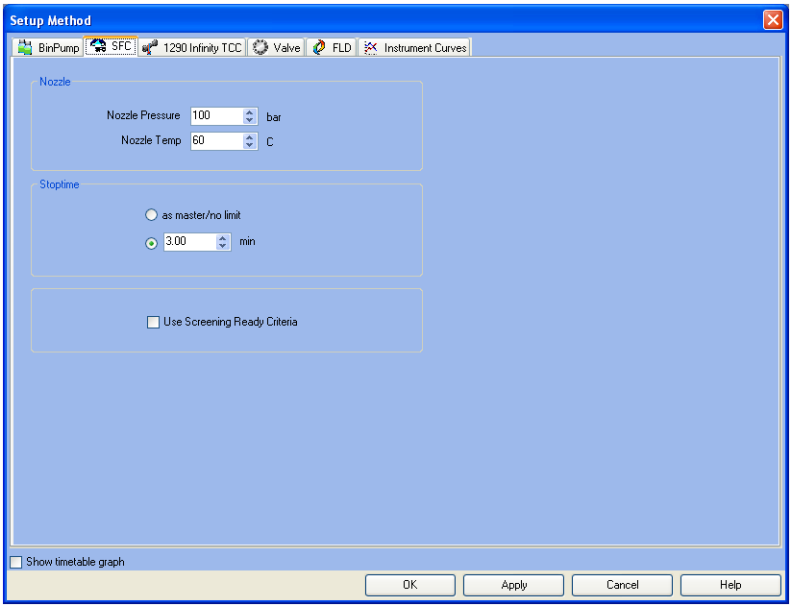
9 Press **OK** to accept the settings.

10 Click **OK** in the Agilent ChemStation **Instrument Configuration** to apply all configurations.

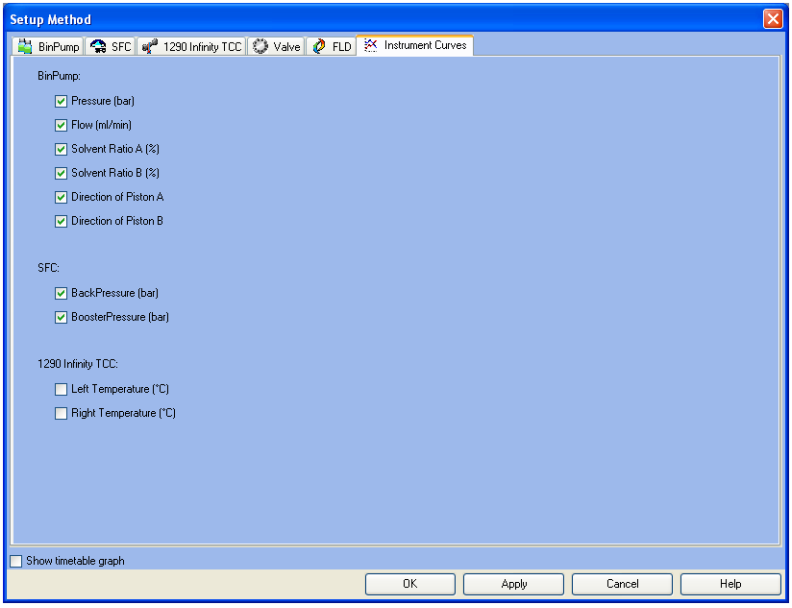


Setting up the Method

1 In the Agilent ChemStation, the method parameters for all RC.Net devices, including SFC settings, are combined into a single tabbed dialog box:



2 The **Instrument Curves** tab shows all instrument curves (monitor traces) available for storing with the acquired data file:



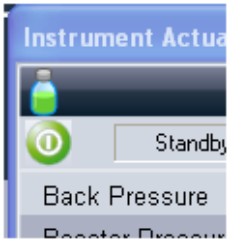
Status

1 To start **Aurora SFC Status** in Agilent ChemStation go to **View > Instrument Actuals**. The **Instrument Actuals** have to be enabled.



2 The available status information depends on the configuration (e.g. Aux Pressure).

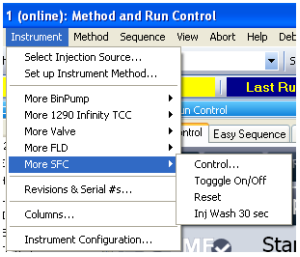
3 The button in the upper left corner of the **Status** user interface allows you to either switch the device ON or put it into Standby mode.



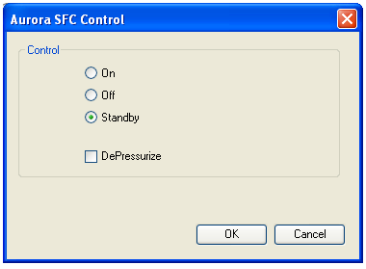
Control

The following steps show you how you can control your SFC instrument in the Agilent ChemStation.

1 In **Instrument > More SFC** you find more menu items that allow you to control the device directly.

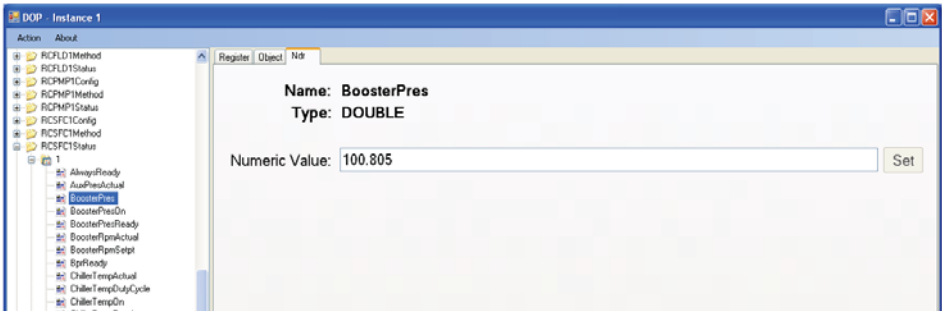


2 To change the status of the SFC device and to Depressurize, go to **Instrument > More SFC > Control**. This displays the **Aurora SFC Control** dialog box.



Testing the System

The SFC driver lists all available configuration, method and status values in ChemStation registers, available by macros.



Additionally, it works with the ChemStation **`SendModule$`**-function. Using this command, you can send commands directly to the SFC module. There are special commands, allowing you to enable and disable diagnostic mode (**`print SendModule$(SFC1`**, **EnableDiag**) / **`print SendModule $`**(**SFC1**, **DisableDiag**).

Enabling **Diagnostic** immediately shows additional status information in the **Status** window. Additionally, a separate configuration menu item shows up in **Instrument > More**, and **Additional instrument curves** are available in the Online plot.

NOTE Enabling **Diagnostic** mode requires a restart of Agilent ChemStation, to show the **Additional configuration** item and **Instrument curves**.



5
Using the Agilent 1260 Infinity SFC Control Module

Powering up the Module 90

Power-up Sequence and Operational Control States 91

Operational Control States 92

- The OFF State 93
- The STANDBY State 93
- The ON State 94

Controlling the Agilent 1260 Infinity SFC Control Module through the Agilent ChemStation 95

Running a method on the SFC system 98

Shutting Down the SFC System 101

- Partial Shutdown 101

This chapter provides information and hints on the use of the SFC System.

Powering up the Module

The Agilent 1260 Infinity SFC Control Module is powered on by pressing the top of the rocker-type power switch located on the upper right rear of the module. Once the rocker is pressed, the module responds by entering its power-up sequence. The power button of the module must remain accessible at all times. Never arrange equipment so that the switch cannot be accessed.

WARNING

The power switch has to be reachable for emergency

➔ The power switch of the module must remain accessible at all times. Never arrange equipment so that the switch cannot be accessed.

Power-up Sequence and Operational Control States

When power is applied to the Agilent 1260 Infinity SFC Control Module, a series of events is initiated. The order of these events is designed to safely initialize and test individual component functionality. The module power-up sequence executes the following steps:

1. Power is automatically applied to the processor and the two module fans.
2. The processor initializes:
   a. Any temporary configuration or calibration data that has not been stored in flash memory is lost.
   b. A checksum validation is made of data stored in flash memory.
   c. A self test is run to test power supply voltage levels and sensor readings for in-range values
   d. Stored calibration and configuration data are downloaded to RAM.
   e. The event logbook is updated
3. The booster pump drive is rotated to find its index pulse.
4. Index pulses are tested for module fans and the coolant pump.
5. The BPR is homed to its fully open (depressurized) position.
6. If installed, the wash pump is rotated to its index pulse.

At the completion of a successful power-up sequence, the processor places the module in the OFF operational state, described in "Operational Control States" on page 92. If an error is encountered and unresolved after multiple attempts, the module is placed into the ERROR state and a notation is stored in the event log.

Operational Control States

The Agilent 1260 Infinity SFC Control Module has three defined operational control states: OFF, STANDBY and ON. You control the three states by selecting the Control option from either the ChemStation SFC Fusion icon, by pressing various icons of the ChemStation GUI, or from the Aurora A5 Diagnostic Program status tab.

Some components of the Agilent 1260 Infinity SFC Control Module are not governed by the three described states but are continuously on. These include:

- ✔ The processor.
  - Continuously records and transmits sensor data to the host control system.
  - Handles status and command requests from the host controller.
  - Monitors sensors for safety-related parameters.
  - Updates the event log.
- ✔ Pressure and temperature sensors are continuously powered and sensed.
- ✔ Coolant and electronics bay fans are continuously powered.
- ✔ The wash pump is activated by contact closure of the external contacted board placed in the SFC Autosampler, independent of the Agilent 1260 Infinity SFC Control Module control state.

The OFF State

The **OFF** state is characterized as follows:

- ✔ The CO_2 supply valve is closed (unpowered).
- ✔ The booster pump drive is unpowered.
- ✔ The BRP drive is unpowered.
- ✔ The BRP heater is unpowered.
- ✔ The chiller is unpowered.
- ✔ The secondary cooling circuit pump is unpowered.

The **OFF** state is always entered after a successful power-up sequence. It can also be entered by selecting the **Off** option in the control window, by a timeout from the STANDBY state or by pressing **Off** in the Agilent ChemStation graphical user interface twice in succession.

When the Agilent 1260 Infinity Control Module is in the **OFF** state, the top "power" status light is constant and the bottom "ready" status light is off.

The STANDBY State

The **STANDBY** state is characterized as follows:

- ✔ The CO_2 supply valve is closed (unpowered).
- ✔ The booster pump drive is unpowered.
- ✔ The BRP drive is powered.
- ✔ The BRP heater is powered.
- ✔ The chiller is powered.
- ✔ The secondary cooling circuit pump is powered.

The **STANDBY** state can also be entered by selecting the **Standby** option in the control window, by pressing the power button associated with the SFC icon of the GUI, or by pressing **Off** in the ChemStation graphical user interface once while the system is running.

When the **STANDBY** state is entered from the OFF state, the BPR must be homed. Once this is accomplished, the BPR drive is active but in a hold state.

When the Agilent 1260 Infinity SFC Control Module is in the **STANDBY** state, the two status lights flash alternately. The **STANDBY** state remains active for up to three hours. If no user-initiated action is taken to change or renew the state within this time, a timeout occurs and the processor automatically enters the **OFF** state.

The ON State

The **ON** state is characterized as follows:

- ✔ The CO_2 supply valve is open (powered).
- ✔ The booster pump drive is powered and begins to pump CO_2 to the pressure target.
- ✔ The BRP drive is powered and the BRP begins the process of regulating to its setpoint.
- ✔ The BRP heater is powered.
- ✔ The chiller is powered.
- ✔ The secondary cooling circuit pump is powered.

The **ON** state can also be entered by selecting the **On** option in the control window, by pressing the power button associated with the SFC icon of the GUI, or by pressing **On** in the ChemStation graphical user interface.

When the Agilent 1260 Infinity SFC Control Module is in the **ON** state, the top status light is continuously lit. The bottom status light is lit when the system reaches a “ready” state, indicating that backpressure and booster pump pressure are under control and stabilized within their control band.

Modules that start from the **OFF** state generally must first perform an initialization routine before moving to **ON**. In the case of the Agilent 1260 Infinity SFC Control Module, initialization causes the BPR first to home, then move to a default initialization position. The booster pump delays operation until the chiller passes below a threshold temperature value.

Controlling the Agilent 1260 Infinity SFC Control Module through the Agilent ChemStation

Exploring the module Graphical User Interface (GUI)



Figure 34 Setting Control State

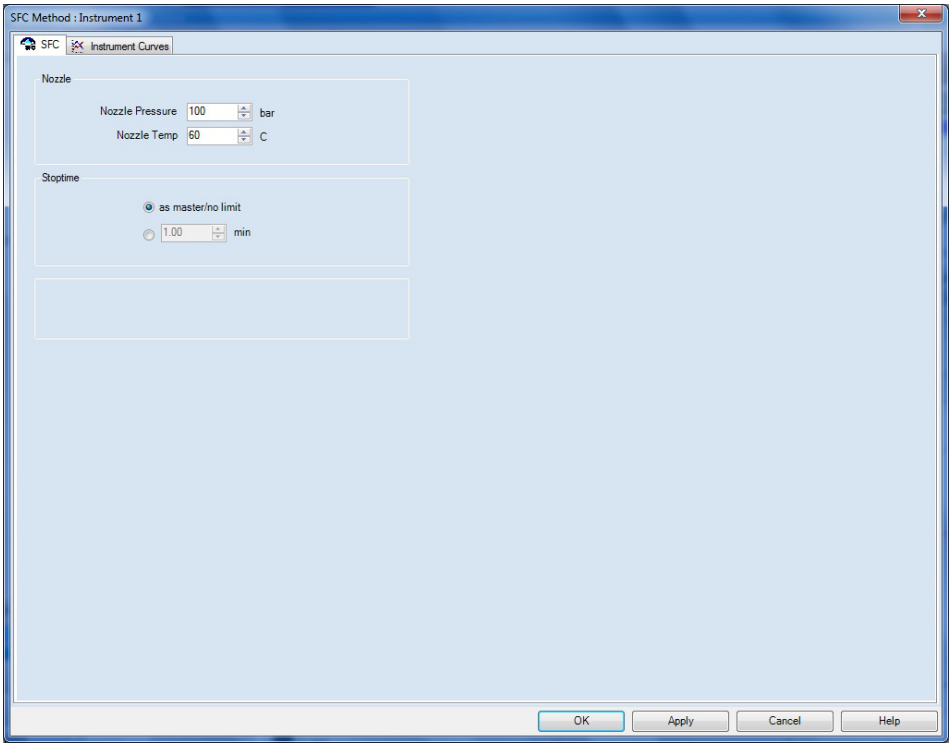
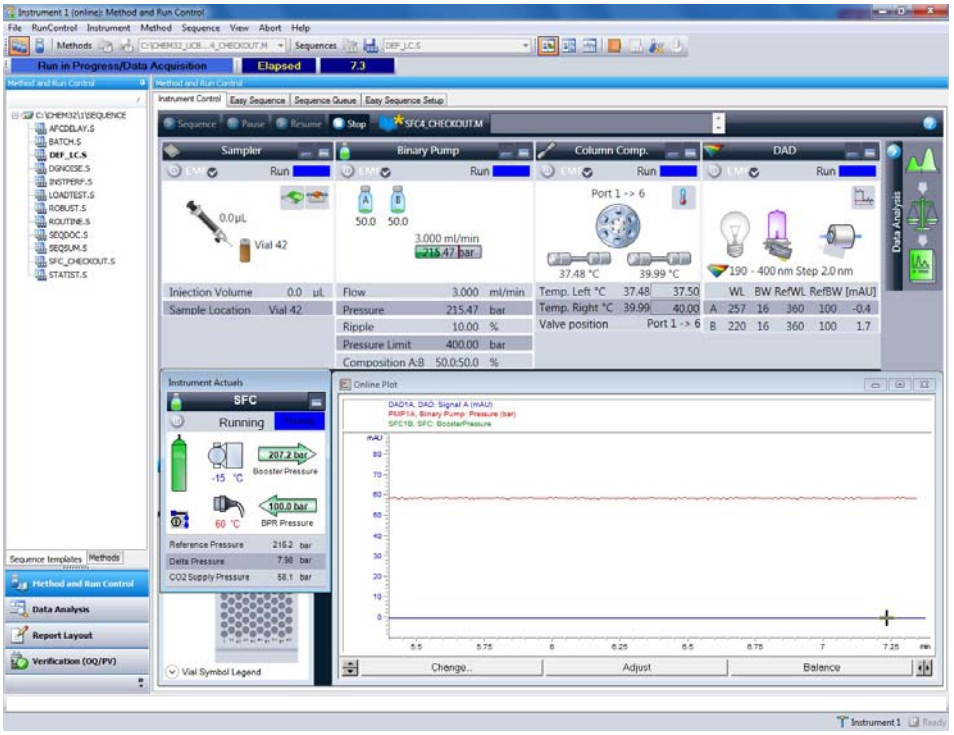


Figure 35 Editing the SFC control module Method Parameters

Editable method parameters for the Agilent 1260 Infinity SFC Control Module are the Nozzle Pressure (system back-pressure) and the Nozzle Temperature (temperature of the back-pressure regulator).

Controlling the wash pump

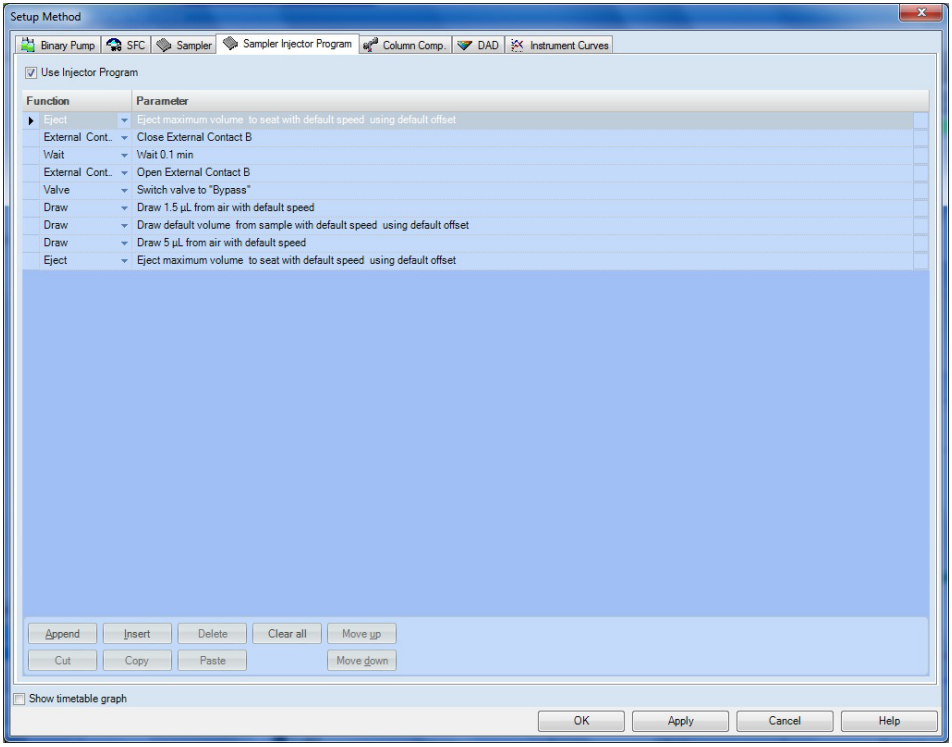


When the wash pump icon is clicked, the wash pump starts pumping for 30 seconds and flushes the autosampler metering, loop, needle and needle seat with solvent, Typically, modifier solvent is used to flush the autosampler. The wash pump is controlled by the Agilent SFC autosampler via the built-in BCD board, and works independently of the current state of the Agilent 1260 Infinity SFC Control Module.

Running a method on the SFC system

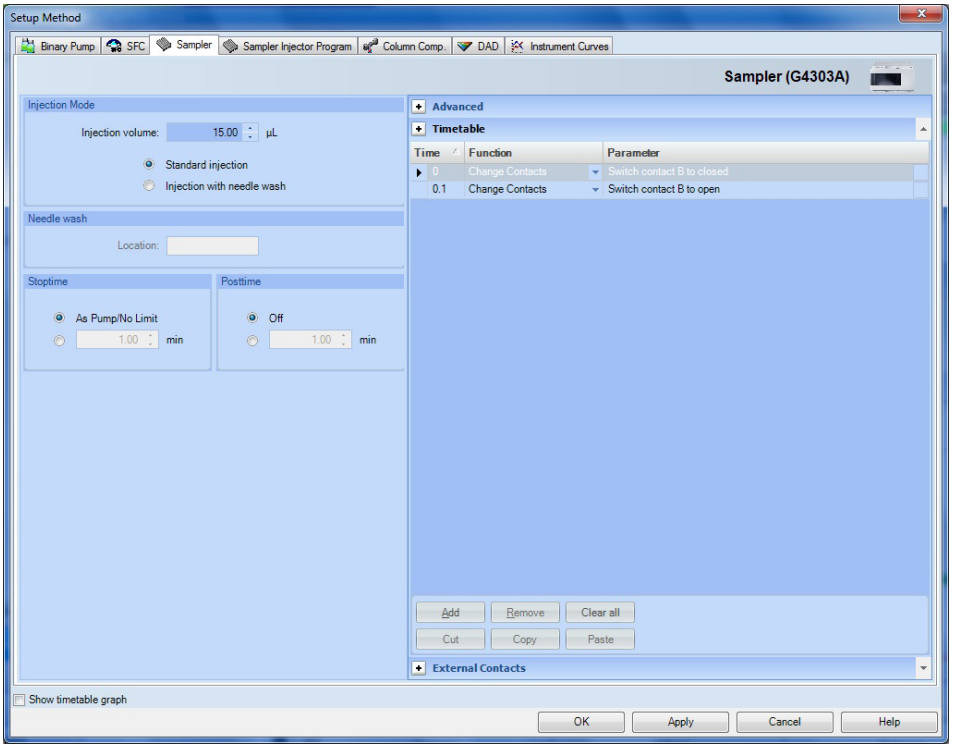
Loading the SFC_Def analysis method

When the SFC Driver add-on for ChemStation is installed, the method `SFC_Def.M` is copied to the default method folder. This method contains parameters most commonly used in SFC. You should develop your SFC methods from this default method.
`SFC_DEF.M` contains an injector program for full loop injection.

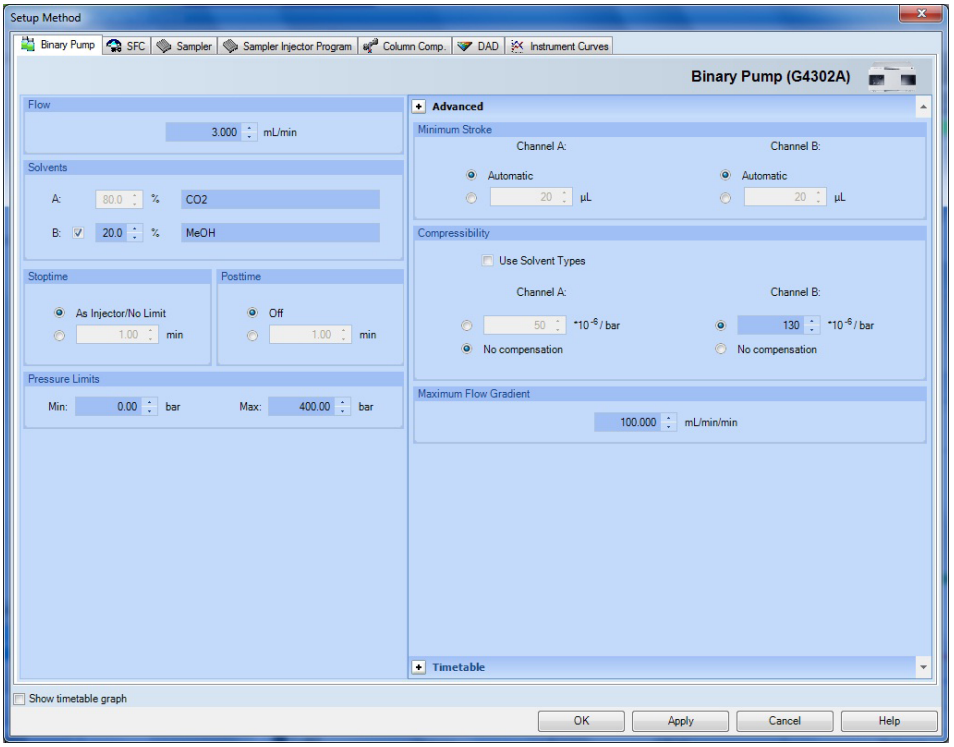


Adjusting the method for use

The Injector timetable includes an additional flush of the Autosampler for 0.1 min by the wash pump. By default, the injection volume is 15 µL, which corresponds to an overfill of the sample loop by approximately three times.



Default Pump setting is 80 % CO_2 : 20 % modifier. Typical Flow rate in SFC is 3 mL/min; however, when `SFC_DEF.M` is loaded, the flow rate is set to 0 mL/min. Solvent compressibility should be always set to **no compensation** for the CO_2 Channel, and to the corresponding value for the modifier.


Setup Method
Binary Pump
SFC
Sampler
Sampler Injector Program
Column Comp.
DAD
Instrument Curves
Binary Pump (G4302A)
Flow
3.000 mL/min
Solvents
A: 80.0 % CO2
B: 20.0 % MeOH
Stoptime
As Injector/No Limit
1.00 min
Posttime
Off
1.00 min
Pressure Limits
Min: 0.00 bar
Max: 400.00 bar
Advanced
Minimum Stroke
Channel A:
Automatic
20 µL
Channel B:
Automatic
20 µL
Compressibility
Use Solvent Types
Channel A:
50 *10-6/bar
No compensation
Channel B:
130 *10-6/bar
No compensation
Maximum Flow Gradient
100.000 mL/min/min
Timetable
Show timetable graph
OK
Apply
Cancel
Help

Shutting Down the SFC System

The manner of shutting down the SFC depends on the requirement for rapid equilibration of the system on the next startup, and the duration of the shutdown. If the system will be shut down for some time, it is probably best to shut down all components including the DAD and column oven. These components tend to take longer to reach their stable operating conditions than the other components in the system. You should always shut down both the SFC control module and the binary pump if the system is to be idle for a long time.

Partial Shutdown

WARNING

Injuries from pressurized CO_2

➔ Setting the pump to STANDBY does not depressurize the system. Do not attempt to loosen fittings or perform maintenance under these conditions. Serious skin and eye injuries can occur as the result of sudden release of CO_2 in the liquid or supercritical state. Always wear gloves and eye protection when maintaining the module.

Leaving the system pressurized

If the system is to be shut down for less than two hours, a partial pressurized shutdown is recommended. Press the **control** buttons (for example, button 2) to switch the Agilent 1260 Infinity SFC Control Module and binary pump states to **STANDBY**. In this case, the system remains pressurized, and slowly bleeds pressure through the nozzle. A residual pressure remains in the system when the nozzle closes fully at lower pressure. The booster remains chilled, and much of the startup CO_2 is preserved. You should be aware that the system is pressurized and not to attempt maintenance under these conditions.

The detector and column oven are left in the ON state, to maintain their readiness. You may elect to exit the ChemStation in this state, and should answer NO to the “Shutdown Lamps...” query that appears during shutdown. Restarting the ChemStation brings the system to this same state. If more than two hours elapse in **STANDBY** mode, the Agilent 1260 Infinity SFC Control Module enters the OFF state, and pressure may be lost at a more rapid rate.

Depressurizing the system

When maintenance is required on the SFC system, such as replacing the column, the system should be depressurized. It is not necessary to shut down all modules, but only the pumps and any other devices undergoing maintenance. To depressurize the system, stop the Agilent 1260 Infinity SFC Control Module and the binary pump. In the **Control...** menu of the Agilent 1260 Infinity SFC Control Module booster (the standby state will be selected) check the **Depressurize** box and click **OK**.

NOTE

If the Agilent 1260 Infinity SFC Control Module unit itself is to be serviced, select OFF in the Control window. This shuts off the BPR heater and booster chiller and allows them to move toward ambient temperature.

This causes the BPR to home and fully open the CO_2 path to depressurize. The Agilent 1260 Infinity SFC Control Module unit contains approximately 25 mL of stored CO_2. This amount of CO_2 should be vented properly, which takes several minutes. You should allow the system to drain below 40 bar before cracking any fittings. At this point the, CO_2 is in the vapor state, and represents a small expanded volume. However, do not inhale vapor directly from a cracked fitting. The concentration of CO_2 emerging from a flow line, even at low pressure, can be dangerous or even lethal.

WARNING

Avoid inhaling high concentration of CO_2

➔ Never inhale vapor issuing from an SFC flow line. Exposure to concentrations of CO_2 over 5 % in air can be lethal. Always keep tubes directed away your face. CO_2 is ubiquitous in the atmosphere, but at high levels should be treated with the same care as other toxic chemicals. Always wear gloves and eye protection for safety. Avoid inhaling venting gas near open fittings.

Alternatively, if the column oven contains a column switching valve, one path may be jumpered without a column. The reduced restriction will allow the system to depressurize much faster. Further, the isolated column can be exchanged immediately since the contained volume of CO_2 is small.

WARNING

System contains always 25 ml liquid CO2 under pressure

➔ The Agilent 1260 Infinity SFC Control Module unit contains approximately 25 mL of liquid CO_2. The CO_2 must be vented properly since the expanded volume will allow local concentrations exceeding the OSHA PEL. Always allow the system to depressurize to below 40 bar before cracking any fittings. Always keep fittings directed away from the face.



6
Maintenance and Repair

Inspection and Preventative Maintenance Intervals 106
- Daily Inspection and Maintenance 106
- Every 3 months 107
- As Needed (Corrective) 107

General Maintenance procedures 108
- Booster Drawer 108
- Replacing Fuses 115
- Standard Decontamination 115
- Plugged BPR Decontamination 116
- Preparing for storage or shipping 117

In this chapter only the SFC specific procedures are described. For procedures similar to the Agilent module procedures, please refer to the single module manuals (G1312C, G1329B, G1316C, G1315/65C, G4225A)

Inspection and Preventative Maintenance Intervals

Inspection and maintenance of the Agilent 1260 Infinity Analytical SFC System are critical elements of long term reliability and performance of the system. Maintenance falls into two categories, preventative and corrective. Preventative maintenance intervals can vary based on the system use. The intervals offered in this section are for systems with average use of approximately 30 hours per week. Infrequently used systems may extend these intervals, while heavily used systems may require more frequent preventative maintenance. Most service can be performed directly by the user or in-house maintenance technicians.

Daily Inspection and Maintenance

- ✔ Verify that power and signal cables are firmly connected and not under strain.
- ✔ Inspect all user-serviceable high pressure tubes and transfer lines for crimping or very tight bends. Replace as necessary.
- ✔ Wipe up any visible liquid spills or condensation on or near the instrument.
- ✔ Verify that all covers are securely fastened to the frame.
- ✔ Inspect all reservoirs to ensure an adequate solvent supply.
- ✔ Prime the wash pump and modifier pumps. Check that the purge valve reseals without leaking
- ✔ Empty all waste containers
- ✔ Check that the exhaust line is attached to a ventilation system, and that the ventilation system is drawing.
- ✔ If an inlet step-down regulator is used with a gas supply system, check that the inlet pressure is between 40 and 70 bar.
- ✔ Check the integrity of the SFC Flow path (that is, that column, flow cell etc have not been removed).
- ✔ With the system running, visually inspect unions and tees for leaks. Run diagnostic leak test for added sensitivity.
- ✔ Check CO_2 air monitor, if available, for suitable exposure level [< 5000 ppm CO_2]

Every 3 months

- ✔ Run nozzle diagnostic test.
- ✔ Run system leak test.
- ✔ Evaluate system calibration.
- ✔ Check chiller efficiency curve.
- ✔ If CO_2 cylinders are used as supply, change the cylinder seal (approximately every 10 cylinders) at the next tank change.
- ✔ Remove visible dust accumulation in the area of the module.

As Needed (Corrective)

- ✔ Change booster and CO_2 pump check valves.
- ✔ Change high pressure transfer lines with metal ferrules or PEEK end fittings after 10-20 reseals or when leaking.
- ✔ Change Booster piston (rare).
- ✔ Exchange BPR head (rare).

General Maintenance procedures

Booster Drawer

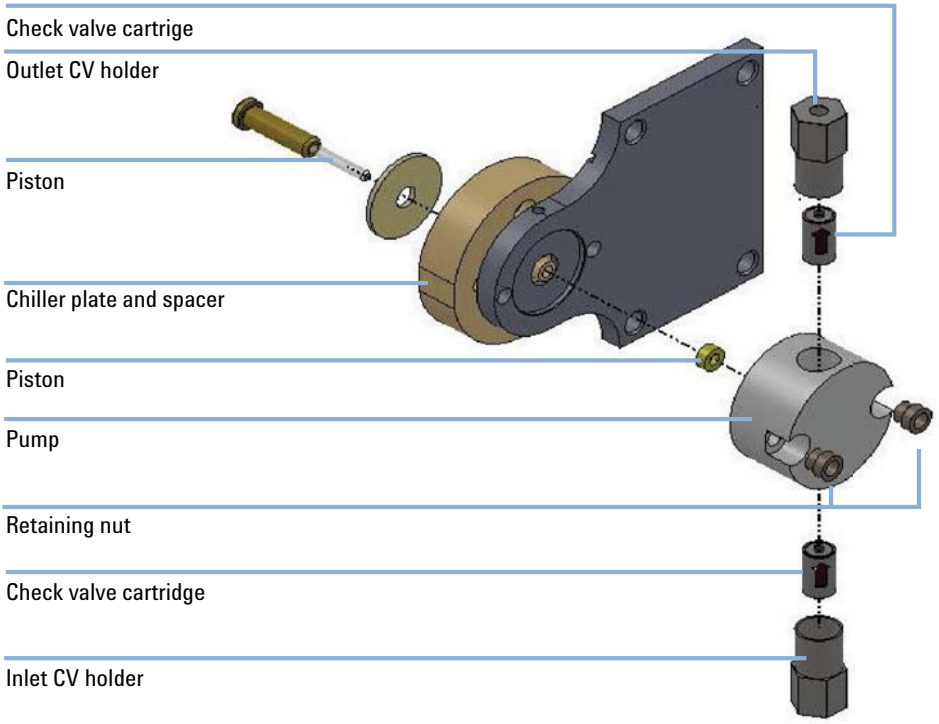


Figure 36 Exploded view of Booster Components

Removing the vapor shield

The vapor shield is not shown in the graphic Figure 36 on page 108.

Most maintenance procedures require removal of the vapor shield to access the underlying pump unit.

Tools required

- A 3/16" hex-drive wrench (mounted on the back of the front cover)

1. Set the Control state to **OFF** on the SFC control module.
2. Wait for the chiller temperature to approach room temperature to prevent significant condensation on the chiller assembly and pump head.
3. Turn the power *OFF* on the SFC control module. Unplug the power cord.
4. Remove the front cover of the SFC control module unit by pulling gently at the upper left and right indents to the rear of the cover. The cover will release from its magnetic catch. Lift the cover upward to clear the two mounting pins at the base and set it aside.

HINT The 3/16" hex wrench used to remove the vapor shield and pump head is stored inside the removable front cover.

5. While holding the vapor shield with one hand, use the 3/16" hex wrench to loosen the four cap screws attaching each corner of the shield until they each disengage from the front panel. The screws are captured in the shield; do not try to remove them completely.
6. Remove the shield and store it in a safe location. Do not use a container for disassembled parts. This will scratch the plastic and impair visibility of the pump head during operation.

Replacing the vapor shield

1. Locate the vapor shield approximately over the mounting holes in the booster drawer front panel.
2. Engage each screw approximately one turn.
3. Inspect the border of the vapor shield to make sure it is in sealing contact with the foam seal of the drawer face. Adjust as necessary.
4. Tighten the mounting screws to ensure at least 50 % compression of the foam seal by the shield.
5. Replace the front cover by aligning the two base mounting pins and tilting forward to engage the magnetic catches.

Replacing Booster Pump check valves

Tools required

- A 1/4″ open-end wrench
- A 9/16″ open-end wrench

Parts required

#	p/n	Description
1	G4301-00200	Plastic cover with screws

1 Remove the vapor shield.

2 Using the 1/4” and 9/16” wrenches, loosen and remover the inlet or outlet capillary tube.

3 Using the 9/16” wrench, loosen and remove the desired check valve holder. The check valve may or may not be extracted with the holder.

4 The inlet check valve assembly includes a PEEK gasket. Set this gasket aside for reuse. (Inlet CV only)

5 Remove the defective check valve cartridge.

CAUTION

Do not mix up check valve directions

→ The orientation of the CV cartridge is critical. An arrow on the side of the cartridge indicates the direction of flow. Make sure the cartridge is installed to allow flow in the proper direction when installed into the pump head.

6 Insert the new check valve cartridge into the holder oriented correctly for the direction of flow (arrow up). The inlet check valve is inserted with the non-filter end of the cartridge showing. The outlet check valve is inserted with the filter end of the cartridge showing.

7 Replace the PEEK gasket on the top of the inlet check valve cartridge with the flat side of the gasket facing the cartridge. (Inlet CV only)

8 Insert the CV holder into the pump head and tighten with a 9/16” wrench.

9 Refasten the inlet or outlet capillary line, holding the CV holder with a 9/16” wrench, and tightening the fitting with the 1/4” wrench to seal.

10 Replace the vapor shield

Removing the Pump head

Tools required

- A 1/4" open-end wrench
- A 9/16" open-end wrench
- A 3/16" hex-drive wrench
- seal insertion/removal tool
- Ultrasonic bath
- Isopropanol
- Deionized water

Parts required

#	p/n	Description
1	G4301-60220	Boaster pump piston kit
1		Piston seal

NOTE Each time the booster pump head is removed, the piston seal should be exchanged, since the seal surface may be easily scratched or distorted during removal. For this reason, the procedures are bundled. Cleaning the pump head is optional after visual inspection. Sealing surfaces of the pump head are critical to successful operation. Never use metal tools or paper toweling to wipe, probe or contact these surfaces.

1 Set the SFC control module mode to **OFF**.

2 Wait for the pump head to reach room temperature.

3 Power off the SFC control module.

4 Remove the vapor shield

5 Using the 9/16" and 1/4" wrenches, remove the inlet line from the inlet check valve holder and the outlet line from the outlet check valve holder.

CAUTION Danger of piston breakage

➔ Be careful not to break the piston when removing the pump head. Twisting the pump head can cause the piston to break.

6 Using the 3/16" hex-drive, carefully remove the two knurled nuts at the front of the pump head.

7 Carefully separate the pump head from the pump. Move the pump head straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the seal from the piston if it did not stay in the pump head.

8 If the seal remains with the pump head, insert the flanged end of the seal insertion/removal tool into the seal cavity. Tilt it slightly so that flange is under the seal and pull out the seal.

Inspecting and Cleaning the Pump Head

1 Visually inspect the piston seal cavity in the pump head. Use magnification if necessary. Remove any foreign material using a cotton swab, or equivalent, and avoid scratching the sealing surfaces. Be sure no fibers from the cleaning swab remain in the components.

2 The pump head, may be further cleaned as follows:

   a Remove inlet and outlet check valves.

   b Clean with 50 % isopropanol in water in an ultrasonic bath for at least 30 min, followed by rinsing for at least 10 min in 100 % isopropanol. Be sure that all particles loosened by the above procedures have been removed from the components before re-assembly.

   c Replace the check valves.

3 Wipe off any residual liquid from external (non-sealing) surfaces with a soft cloth such as a microfiber towel.

Replacing the Piston Seal

1 Sonicate or soak the new seal in isopropanol for 15 min to clean and provide lubrication for installing.

2 Place the replacement seal on the rod-shaped end of the seal insertion/removal tool so that the spring is visible when the seal is fully seated on the tool.

3 Insert the tool into the pump head so that the open side of the seal enters first, facing the high-pressure cavity of the pump head.

4 Be careful to line up the seal with the cavity while inserting. Withdraw the tool, leaving the seal in the pump head.

   When you look into the pump head cavity, only the polymer portion of the seal should be visible.

Replacing the Pump Head

1 Fill the pump head cavity about one third full with isopropyl alcohol.

2 Wet the piston tip with a few drops of isopropyl alcohol.

3 Holding an absorbent towel beneath the pump head assembly, line up the pump head and carefully slide it into place. Be sure that the inlet valve is on the bottom and the outlet valve is on the top. Do not force the pump head into place.

4 Finger tighten both knurled nuts into place. To tighten firmly, alternately turn nuts 1/4 turn while gently wiggling the pump head to center it.

5 Re-attach the inlet and outlet lines.

Cleaning or Replacing Booster Pump Piston

Tools required

- Tools for removing the vapor shield (see "Removing the vapor shield" on page 109) and pump head (see "Removing the Pump head" on page 111)
- A 9/64" hex-drive wrench

NOTE

In most cases, this procedure will be used only to replace a broken piston. Pumping CO_2 does not tend to leave deposits on the piston. Development of such deposits warrants examination of the CO_2 supply system and correcting the source of the deposited materials. Release of extraneous materials into the CO_2 supply system may cause contamination of the Agilent 1260 Infinity Analytical SFC System.

1 Remove the Vapor Shield.

2 Remove the pump head.

3 Clean the pump head.

CAUTION

Take care not to break coolant tubes

➔ Use care removing the chiller assembly from the mounting posts. The assembly is connected to a circulation pump behind the drawer panel. Do not pull the flow lines hard as this may loosen or crimp the tubes and cause the chiller to lose efficiency or cause leaks in the secondary cooler system.

4 With a gentle rocking motion, loosen the chiller plate assembly and carefully slide it forward off the pump head mounting posts. Carefully twist the assembly out of the way.

5 Use the 9/64” hex wrench to unscrew the two cap screws attaching the spacer and very carefully remove the spacer by pulling straight back. This fully exposes the piston and retaining ring.

6 Remove the retaining ring by prying it out with a small blunt instrument or tweezers at the slot provided.

7 Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up.

8 Grasp the metal base of the replacement piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot.

9 Replace the retaining ring and spacer. Reattach the spacer mounting screws. If properly positioned, the spacer should be pressed into the foam wall seal.

10 Gently slide the chiller back onto the pump mounting posts and firmly press it onto the spacer. If properly positioned, the chiller heat exchanger should now be pressed into the foam wall seal.

11 Replace the piston seal

12 Replace the pump head

13 Replace the vapor shield

Replacing the CO_2 Inlet Filter

1 Unscrew the filter closure from the filter housing.

2 Use a seal insertion/removal tool or a non-metallic object (such as a wooden toothpick) to remove the large seal that remains in the housing

3 Unscrew the old filter and remove the small seal from the filter closure.

4 Place one of the small seals included in the replacement element kit over one of the new filters from the kit. Screw the new filter into the filter closure (finger tight).

5 Place one of the large seals from the replacement kit on the filter closure. Insert the filter closure into the housing and tighten.

Replacing Fuses

The power entry module of the Agilent 1260 Infinity SFC Control Module unit contains an external fuse drawer that is user serviceable.

To replace fuses: (before replacing fuses, first try to determine cause of fuse activation and repair)

1. Power down the unit.
2. Disconnect the power cable from the power entry module.
3. Depress the release lever of the fuse drawer and pull the drawer straight back to remove.
4. Replace blown fuses with 8 A250 V Time Delay fuses of matching size. (A set of replacement fuses is included shipping kit).
5. Replace the fuse drawer by sliding it into the power entry module until it locks into place.

Standard Decontamination

Cleaning

External surfaces of the enclosure can be wiped with a damp soft cloth. More stubborn marks can be removed with a 50 % isopropanol:water mixture or mild cleanser such as Soft Scrub™. The latter may also be used to remove surface paint blemishes that may result from normal use.

The vapor shield of the booster drawer should be wiped only with a very soft cloth such as a microfiber polypropylene cloth, otherwise the surface may be scratched. Other user-accessible internal surfaces can be cleaned with a damp cloth.

BPR

The BPR head contacts CO_2, modifiers and sample material. To decontaminate, rinse with 50 % modifier flow at 5 mL/min for 15 min followed by pure CO_2 for 5 min.

Wash Pump

1 Drain the inlet line of old solvent.

2 Flush the inlet line and filter from a small intermediate reservoir to rinse contaminated residual fluid from the lines.

3 Insert the inlet line into a fresh supply of pure solvent

4 Prime the wash pump for 2 min (four consecutive presses of the 30 s timer) to clear the remaining flow path.

Plugged BPR Decontamination

Decontamination of plugged BPR heads may require more aggressive solvents. In this case use the following procedure:

1 Depressureize the SFC control module unit completely.

2 Disconnect the BPR inlet and outlet tubes from the BPR drawer.

3 Attach the Inlet tube via a transfer line to waste.

4 Attach a solvent pump to the outlet tube of the BPR head

5 Prime the pump with a suitable solvent for the obstructing material.

6 Flush backwards with strong solvent at 1 mL/min for 20 minutes. Do not exceed a pressure of 400 bar.

7 If the pump cannot transfer fluid at less than 400 bar discontinue the operation and perform steps to exchange the BPR head.

8 If the backflush is successful, rinse the BPR head with Isopropanol for 10 minutes at 1 mL/min to clear the strong solvent.

9 Reconnect the BPR inlet and outlet lines.

10 Complete the standard decontamination procedure listed above

Preparing for storage or shipping

If the SFC control module needs to be stored in other than its operational location, it is best to store it in the original factory packaging. This packaging can also be used to reship the device to a secondary location. If the original packaging is unavailable, the unit should be stored upright and preferably covered in a plastic bag or wrap to prevent exposure to dust.

To prepare the unit for storage use the following procedure:

1. Follow the standard decontamination procedure.
2. Depressurize the SFC system completely.
3. Power off the unit.
4. Remove the front panel.
5. Disconnect the wash pump transfer line from the autosampler.
6. Drain the wash pump lines of fluid.
7. Coil the lines to fit in the SFC control module behind the removable front panel.
8. Disconnect the booster pump transfer line from the binary pump at the pump inlet check valve.
9. Disconnect the BPR return line from the detector.
10. Coil both lines to fit inside the SFC control module behind the removable front panel.
11. Replace the front panel
12. Disconnect the power cord and all signal cables from both ends of the connection.
13. Store cables and cords in a large plastic zip-lock bag.
14. Cover the unit with a large plastic bag.
15. If the original container is available, place the unit with its left side down in the packaging. Otherwise, store the unit upright in the storage area.
16. If the HPLC will also be stored or shipped, and will be reconfigured as an SFC system, the upgrade components can remain in the system.
17. If the two systems are to be permanently separated, uninstall the check valves, modifier purge valve, 3-groove rotor and high pressure flow cell by reversing the installation procedures in "Hardware Installation" on page 40. Store the components along with the original software disc and any upgrades with the module.



7
Parts for Maintenance

Agilent 1260 Infinity SFC Control Module Parts 120
Agilent Module Parts 121

This chapter provides information on parts for maintenance and repair.

Agilent 1260 Infinity SFC Control Module Parts

p/n	Description
G4301-60350	Tee (Valco)
G4301-60260	Booster Pump Head
G4301-60250	A5 Pump Seals, 2pk
G4301-60140	Wash Pump Seal Kit
G4301-60130	Wash Pump Piston Assy
G4301-60210	Check Valve Cartridge; 0.125 ball
G4301-60230	Check Valve Holder (A5)
G4301-60120	Wash Pump Check Valve PKG OF 2
G4301-60240	Check Valve Gasket Inlet, 5pk
G4301-60241	Check Valve Gasket Outlet
G4301-60300	CO2 Supply Line
G4301-60310	Pump Transfer Line
G4301-60320	Return Transfer Line
G4301-60150	Wash Pump Transfer Line
G4301-60160	Wash Pump Transfer Line (EVO)
G4301-60560	Cross and Cap. Kit for Aux Pres.Sensor
G4301-60340	Pressure sensor Witness line short
G4301-60330	Pressure sensor Witness line long
0100-2606	Manifold 6 to 1 for Column screening
G4301-00200	Thermal Isolation Cover Assy
G4301-60220	Booster Pump Piston Kit
G4301-60500	Heated Nozzle Assembly

Agilent Module Parts

Agilent 1260 Infinity SFC Binary Pump

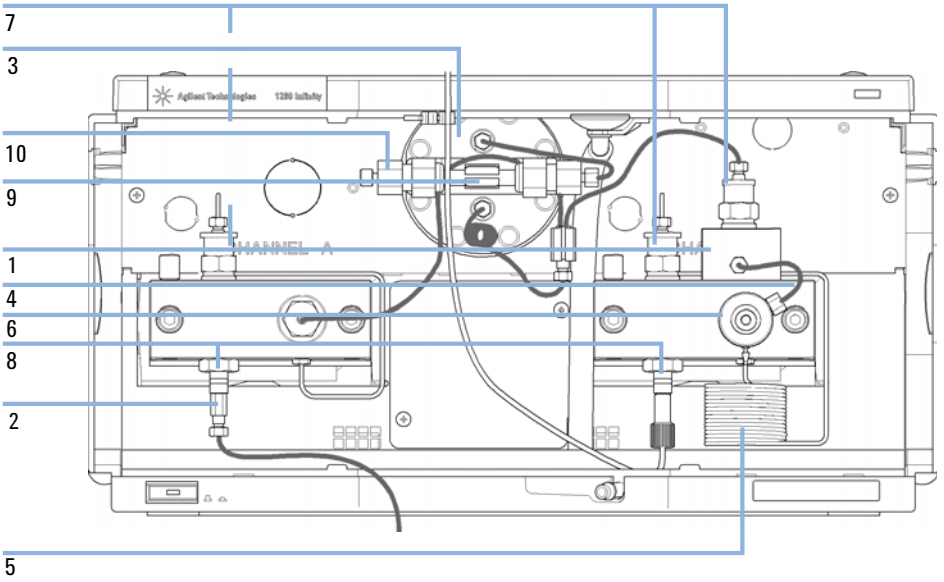


Figure 37 Agilent 1260 Infinity SFC Binary Pump specific SFC Parts

p/n	Description
G4302-20000	Adapter OV SFC
5023-1803	Adapter male/female
G1311-60065	Damper (600bar with pressure Sensor)
G1312-87306 (2x)	Capillary 105x0.17 mm
G1312-87300	Absorber capillary
G1312-60061	Purge valve
G1312-60067	Outlet check valve
G1312-60066	Passive inlet valve
G1312-04100	Mixer Clamp
G1312-87330	Mixer
0905-1420	Pump seals, PE (PumpHead A)
0905-1503	Pump seals, PTFE (PumpHead B)

NOTE For all other Part Numbers please refer to the G1312B User manual.

Agilent 1260 Infinity SFC Autosampler

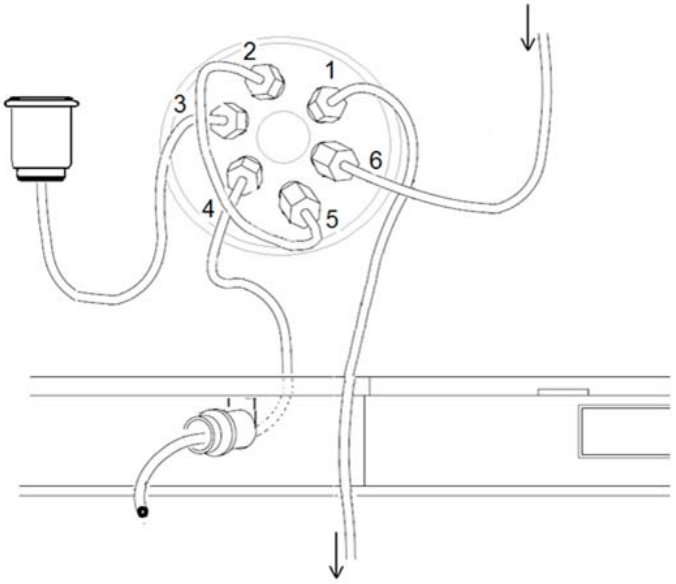


Figure 38 Autosampler Valve

p/n	Description
0101-1409	3 Groove rotor seal
0101-1248	Sample Loop 5µl

NOTE For all other Part Numbers please refer to the G1329B User manual.

DAD SFC Parts

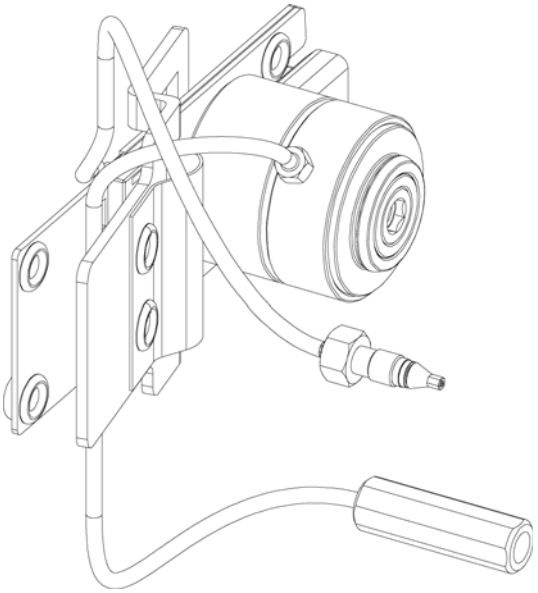


Figure 39 DAD Flowcell SFC

p/n	Description
G4301-60100	SFC Flow Cell
79883-68700	High Pressure Flow cell refurbishment Kit

NOTE For all other Part Numbers please refer to the G1315/65C User manual.

8
Identifying Cables

SFC Control Module Cables 126
Overview 127
BCD Cables 128
External Contact Cable 130
CAN/LAN Cables 131
RS-232 Cables 132

This chapter provides information on cables used with the Agilent 1200 Infinity Series modules.

SFC Control Module Cables

p/n	Description
00-84-2040	Relay Cable, HD15M/F (VGA type), 6'
00-84-2020	BNC Cable
00-84-2030	Remote Cable, DB9 M/M
00-84-2090	USB Cable - 6'

Overview

NOTE Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

BCD cables

p/n	Description
G1351-81600	Agilent module to general purpose

CAN cables

p/n	Description
5181-1516	CAN cable, Agilent module to module, 0.5 m
5181-1519	CAN cable, Agilent module to module, 1 m

RS-232 cables

p/n	Description
G1530-60600	RS-232 cable, 2 m

LAN cable

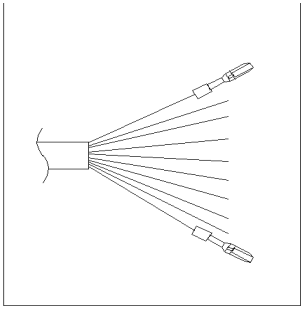
p/n	Description
5023-0203	Cross-over network cable, shielded, 3 m (for point to point connection)
5023-0202	Twisted pair network cable, shielded, 7 m (for point to point connection)

BCD Cables

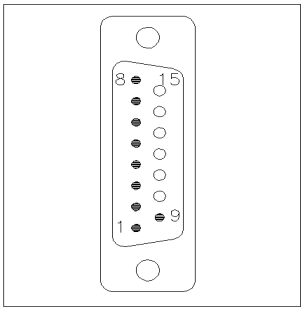


One end of these cables provides a 15-pin BCD connector to be connected to the Agilent modules. The other end depends on the instrument to be connected to

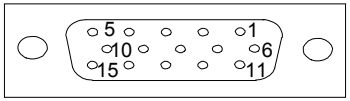
Agilent Module to General Purpose

p/n G1351-81600	Wire Color	Pin Agilent module	Signal Name	BCD Digit
	Green	1	BCD 5	20
	Violet	2	BCD 7	80
	Blue	3	BCD 6	40
	Yellow	4	BCD 4	10
	Black	5	BCD 0	1
	Orange	6	BCD 3	8
	Red	7	BCD 2	4
	Brown	8	BCD 1	2
	Gray	9	Digital ground	Gray
	Gray/pink	10	BCD 11	800
	Red/blue	11	BCD 10	400
	White/green	12	BCD 9	200
	Brown/green	13	BCD 8	100
	not connected	14		
	not connected	15	+ 5 V	Low

Agilent Module to 3396 Integrators

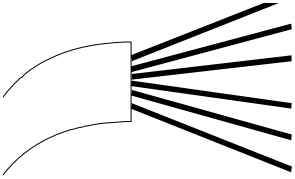
p/n 03396-60560	Pin 3396	Pin Agilent module	Signal Name	BCD Digit
	1	1	BCD 5	20
	2	2	BCD 7	80
	3	3	BCD 6	40
	4	4	BCD 4	10
	5	5	BCD0	1
	6	6	BCD 3	8
	7	7	BCD 2	4
	8	8	BCD 1	2
	9	9	Digital ground	
	NC	15	+ 5 V	Low

External Contact Cable

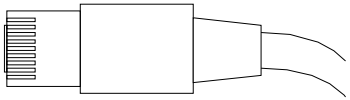


One end of this cable provides a 15-pin plug to be connected to Agilent modules interface board. The other end is for general purpose.

Agilent Module Interface Board to general purposes

p/n G1103-61611	Color	Pin Agilent module	Signal Name
	White	1	EXT 1
	Brown	2	EXT 1
	Green	3	EXT 2
	Yellow	4	EXT 2
	Grey	5	EXT 3
	Pink	6	EXT 3
	Blue	7	EXT 4
	Red	8	EXT 4
	Black	9	Not connected
	Violet	10	Not connected
	Grey/pink	11	Not connected
	Red/blue	12	Not connected
	White/green	13	Not connected
	Brown/green	14	Not connected
	White/yellow	15	Not connected

CAN/LAN Cables



Both ends of this cable provide a modular plug to be connected to Agilent modules CAN or LAN connectors.

CAN Cables

p/n	Description
5181-1516	CAN cable, Agilent module to module, 0.5 m
5181-1519	CAN cable, Agilent module to module, 1 m

LAN Cables

p/n	Description
5023-0203	Cross-over network cable, shielded, 3 m (for point to point connection)
5023-0202	Twisted pair network cable, shielded, 7 m (for point to point connection)

RS-232 Cables

p/n	Description
G1530-60600	RS-232 cable, 2 m
RS232-61601	RS-232 cable, 2.5 m Instrument to PC, 9-to-9 pin (female). This cable has special pin-out, and is not compatible with connecting printers and plotters. It's also called "Null Modem Cable" with full handshaking where the wiring is made between pins 1-1, 2-3, 3-2, 4-6, 5-5, 6-4, 7-8, 8-7, 9-9.
5181-1561	RS-232 cable, 8 m



9 Appendix

General Safety Information 134

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002-96-EC) 137

Radio Interference 138

Sound Emission 139

Solvent Information 140

Agilent Technologies on Internet 141

This chapter provides addition information on safety, legal and web.

General Safety Information

Safety Symbols

Table 15 Safety Symbols

Symbol	Description
	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect risk of harm to the operator and to protect the apparatus against damage.
	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	Indicates eye damage may result from directly viewing the light produced by the deuterium lamp used in this product.
	The apparatus is marked with this symbol when hot surfaces are available and the user should not touch it when heated up.

WARNING

A WARNING

alerts you to situations that could cause physical injury or death.

➔ Do not proceed beyond a warning until you have fully understood and met the indicated conditions.

CAUTION

A CAUTION

alerts you to situations that could cause loss of data, or damage of equipment.

➔ Do not proceed beyond a caution until you have fully understood and met the indicated conditions.

General Safety Information

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

WARNING

Ensure the proper usage of the equipment.

The protection provided by the equipment may be impaired.

➔ The operator of this instrument is advised to use the equipment in a manner as specified in this manual.

Safety Standards

This is a Safety Class I instrument (provided with terminal for protective earthing) and has been manufactured and tested according to international safety standards.

Operation

Before applying power, comply with the installation section. Additionally the following must be observed.

Do not remove instrument covers when operating. Before the instrument is switched on, all protective earth terminals, extension cords, auto-transformers, and devices connected to it must be connected to a protective earth via a ground socket. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. Whenever it is likely that the protection has been impaired, the instrument must be made inoperative and be secured against any intended operation.

Make sure that only fuses with the required rated current and of the specified type (normal blow, time delay, and so on) are used for replacement. The use of repaired fuses and the short-circuiting of fuse holders must be avoided.

Some adjustments described in the manual, are made with power supplied to the instrument, and protective covers removed. Energy available at many points may, if contacted, result in personal injury.

Any adjustment, maintenance, and repair of the opened instrument under voltage should be avoided whenever possible. When inevitable, this has to be carried out by a skilled person who is aware of the hazard involved. Do not attempt internal service or adjustment unless another person, capable of rendering first aid and resuscitation, is present. Do not replace components with power cable connected.

Do not operate the instrument in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a definite safety hazard.

Do not install substitute parts or make any unauthorized modification to the instrument.

Capacitors inside the instrument may still be charged, even though the instrument has been disconnected from its source of supply. Dangerous voltages, capable of causing serious personal injury, are present in this instrument. Use extreme caution when handling, testing and adjusting.

When working with solvents, observe appropriate safety procedures (for example, goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet by the solvent vendor, especially when toxic or hazardous solvents are used.

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002-96-EC)

Abstract

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC), adopted by EU Commission on 13 February 2003, is introducing producer responsibility on all Electric and Electronic appliances from 13 August 2005.

NOTE



This product complies with the WEEE Directive (2002/96/EC) marking requirements. The affixed label indicates that you must not discard this electrical/electronic product in domestic household waste.

Product Category: With reference to the equipment types in the WEEE Directive Annex I, this product is classed as a “Monitoring and Control instrumentation” product.

Do not dispose off in domestic household waste

To return unwanted products, contact your local Agilent office, or see www.agilent.com for more information.

Radio Interference

Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

Test and Measurement

If test and measurement equipment is operated with equipment unscreened cables and/or used for measurements on open set-ups, the user has to assure that under operating conditions the radio interference limits are still met within the premises.

Sound Emission

Manufacturer's Declaration

This statement is provided to comply with the requirements of the German Sound Emission Directive of 18 January 1991.

This product has a sound pressure emission (at the operator position) < 70 dB.

- Sound Pressure Lp < 70 dB (A)
- At Operator Position
- Normal Operation
- According to ISO 7779:1988/EN 27779/1991 (Type Test)

Solvent Information

Flow Cell

To protect optimal functionality of your flow-cell:

- Avoid the use of alkaline solutions (pH > 9.5) which can attack quartz and thus impair the optical properties of the flow cell.

Use of Solvents

Observe the following recommendations on the use of solvents.

- Brown glass ware can avoid growth of algae.
- Avoid the use of the following steel-corrosive solvents:
  - Solutions of alkali halides and their respective acids (for example, lithium iodide, potassium chloride, and so on),
  - High concentrations of inorganic acids like sulfuric acid and nitric acid, especially at higher temperatures (if your chromatography method allows, replace by phosphoric acid or phosphate buffer which are less corrosive against stainless steel),
  - Halogenated solvents or mixtures which form radicals and/or acids, for example:

    $2CHCl_3 + O_2 \rightarrow 2COCl_2 + 2HCl$

    This reaction, in which stainless steel probably acts as a catalyst, occurs quickly with dried chloroform if the drying process removes the stabilizing alcohol,
  - Chromatographic grade ethers, which can contain peroxides (for example, THF, dioxane, di-isopropylether) such ethers should be filtered through dry aluminium oxide which adsorbs the peroxides,
  - Solvents containing strong complexing agents (e.g. EDTA),
  - Mixtures of carbon tetrachloride with 2-propanol or THF.

Agilent Technologies on Internet

For the latest information on products and services visit our worldwide web site on the Internet at:

http://www.agilent.com

Index

A

Agilent
on internet 141
algae 140
ambient non-operating temperature 33, 34, 35, 36, 37
ambient operating temperature 33, 34, 35, 36, 37

B

BCD
cable 128, 127

C

cable
BCD 128, 127
CAN 131
external contact 130
LAN 131, 127
RS-232 132, 127
cables
overview 127
CAN
cable 131
condensation 27

D

dimensions 33, 34, 35, 36, 37

E

electrostatic discharge (ESD) 69
external contact
cable 130

F

flow cell 140
solvent information 140
frequency range 33, 34, 35, 36, 37

H

humidity 33, 34, 35, 36, 37

I

installation
power cords 25
site requirements 24
internet 141

L

laboratory bench 26
LAN
cable 131, 127
line frequency 33, 34, 35, 36, 37
line voltage 33, 34, 35, 36, 37

N

non-operating altitude 33, 34, 35, 36, 37
non-operating temperature 33, 34, 35, 36, 37

O

operating Altitude 33, 34, 35, 36, 37
operating temperature 33, 34, 35, 36, 37

P

physical specifications 33, 34, 35, 36, 37
power consideration 24
power consumption 33, 34, 35, 36, 37
power cords 25

R

RS-232C
cable 132
RS-232
cable 127

S

safety class I 135
safety
general information 135
standards 35, 34, 37, 33, 36
symbols 134
site requirements 24
solvents 140
specification
physical 37, 34, 35, 33, 36
Specifications 28

V

vacuum degasser 36
voltage range 33, 34, 35, 36, 37

W

weight 33, 34, 35, 36, 37

www.agilent.com

In This Book

The manual describes the following:

- Introduction
- Site Requirements and Specifications
- Installing the System
- Configuring the System
- Using the Agilent 1260 Infinity SFC Control Module
- Maintenance and Repair
- Parts for Maintenance
- Identifying Cables
- Appendix

© Agilent Technologies 2010 - 2012

Printed in Germany
12/2012

G4301-90001

Agilent Technologies